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<b>(54) Title:</b> BREATHABLE COMPOSITE SHEET STRUCTURE  <b>(57) Abstract</b> <p>A moisture vapor permeable, substantially liquid impermeable composite sheet material (10) comprising a fibrous substrate (14, 16) and a moisture vapor permeable thermoplastic film layer (12). The moisture vapor permeable film has an average thickness of less than 25 microns, a peel strength of at least 0.1 N/cm, a dynamic fluid transmission of less than about 0.75 g/m<sup>2</sup> when subjected to an impact energy of about 2400 joules/m<sup>2</sup>, a hydrostatic head of at least 60 cm, and a moisture vapor transmission rate, according to the desiccant method, of at least 2800 g/m<sup>2</sup>/24hr. The moisture vapor permeable film is preferably comprised of at least about 50 % by weight of polymer selected from the group of block copolyether esters, block copolyether amides, polyurethanes, polyvinyl alcohol, and combinations thereof. A method for making such a sheet material is also provided.</p> <div data-bbox="820 1134 1242 1344" style="text-align: center;"> </div>		

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TITLE  
BREATHABLE COMPOSITE SHEET STRUCTURE

Field of the Invention

5           This invention relates to a moisture vapor permeable, substantially liquid impermeable composite sheet structure useful in apparel, surgical drapes, sterile wraps, packaging materials, protective covers, construction materials, and personal care absorbent articles such as diapers and sanitary napkins. More particularly, the invention is directed to a thin moisture vapor permeable film and  
10   a fibrous substrate that combine to form a composite sheet that is durable, strong, and flexible, that acts as a barrier to liquids, bacteria, viruses and odors, yet is also highly permeable to moisture vapor.

Background of the Invention

15           Sheet materials used in making medical drapes, medical gowns and absorbent articles, such as diapers and sanitary napkins, must be comfortable, soft, pliable and substantially liquid impermeable. Manufacturing and use requirements for such products often demand that the sheet material also be strong and durable. The sheet materials used in medical apparel and absorbent articles  
20   function to contain the discharged materials and/or to isolate these materials from the body of the wearer or from the wearer's garments and bed clothing. An ideal sheet material for use in medical apparel and absorbent articles will exhibit a high moisture vapor transmission rate that will reduce the build up of heat and humidity inside garments and articles made from the material. The ideal sheet  
25   material will also exhibit excellent barrier properties so as to prevent the passage or seepage of fluids, and will even prevent the passage of bacteria and viruses. Finally, the ideal material must be strong enough so that it does not rip or delaminate under normal usage conditions regardless of whether the material is dry or wet.

30           It is known that the exterior of the sheet material used in medical apparel and absorbent articles can comprise a flexible, fluid and vapor impervious cover to prevent fluids from passing through garments and articles made from the sheet material. These outer covers are often constructed from fluid and vapor impervious films such as polyethylene. While these films do an admirable job of  
35   containing liquids, they are not pleasing to the touch and they do not readily pass moisture vapor, which makes garments and articles made with such films uncomfortable and irritating to the wearer.

Certain polymeric films have been made more acceptable for apparel and personal care applications by creating micropores in the films to make breathable (*i.e.*, moisture vapor permeable) microporous films. In microporous films, moisture is transported through the films by way of small gaps or holes in the film. One notable microporous film composite is made from  
5 polytetrafluoroethylene that is adhered to a textile material with an adhesive, as disclosed in British Patent Application No. 2,024,100. Microporous films adhesively bonded to textile substrates have been used in a variety of apparel products, including absorbent articles, as disclosed in PCT Patent Publication  
10 Nos. WO 95/16562 and WO 96/39031.

Laminates of a microporous film and a fibrous textile substrate have a number of disadvantages, including that such laminates permit some seepage of fluids when used as the backsheet in an absorbent article. For example, when microporous film laminates are used as the backsheet of a disposable diaper, the  
15 backsheet may permit the transmission of some urine through the pores in the backsheet when an infant wearing the diaper sits down. Liquid seepage through microporous film laminates is especially likely to occur when the microporous laminate is exposed to a fluid with a low surface tension, as for example when urine in a diaper is exposed to surfactants within the diaper itself. In addition,  
20 liquid seepage worsens as moisture vapor transmission increases. This is a result of an increase in pore size or in the actual number of pores.

When fluids seep through the pores of a microporous film, bacteria, viruses, and other microbes can pass through the film along with the fluids. Likewise, the passage of fluids through laminates made with microporous films,  
25 whether the fluids are liquid or gaseous, also increases the odors that emanate from such laminates. Microbial adsorbents have been added to some microporous films in an attempt to capture microbes passing through such films, as disclosed in PCT Patent Publication No. WO 96/39031. However, it is difficult to distribute microbial adsorbents throughout a microporous film in a manner that will adsorb  
30 all microbes seeping through the holes in the film. Likewise, microbial adsorbents are unlikely to prevent the passage of odors through the pores in a microporous film.

Moisture vapor permeable films comprised of polyether block copolymers, like the film disclosed in U.S. Patent No. 4,493,870, have an  
35 advantage in apparel and personal care applications because such films are non-porous and therefore substantially impermeable to fluids, but they permit the passage of moisture vapor. U.S. Patent Nos. 4,725,481; 5,422,172; and 5,445,874 disclose that moisture vapor permeable polyether block copolymer

films can be attached to a variety of fibrous substrates including polyester, polypropylene and nylon. Bonding methods used to join the polyether block copolymer films to the fibrous substrates include adhesive lamination, thermal lamination and extrusion coating. Adhesive lamination and thermal lamination are generally carried out in a two step process whereby the film is first formed and is subsequently laminated to the fibrous substrate. With extrusion coating, a melted film is extruded directly onto a fibrous substrate and then passed through a nip while the film is still hot in order to press the film into engagement with the fiber network of the fibrous sheet.

Adhesive lamination, thermal lamination and extrusion coating methods have all been used to produce composite sheets of a fibrous nonwoven substrate and a moisture vapor permeable, substantially liquid impermeable film. It has been possible to make such composite sheets with good barrier properties so long as the moisture vapor permeable film is relatively thick (*i.e.*, > 25 microns). However, it has not been possible to make such composite sheets with thinner films without sacrificing important barrier properties. Very thin moisture vapor permeable films are desirable in a composite sheet because thinner films facilitate significantly greater flux of moisture vapor through the composite sheet and because thinner films use less of the film material and are accordingly less expensive to produce.

Adhesive lamination is carried out in a post film formation step. For adhesive lamination to be feasible, the moisture vapor permeable film must have enough structure, tensile strength and tear strength such that the film can be formed, wound onto a roll, and later unwound and handled during the adhesive lamination process. It is extremely difficult to handle moisture vapor permeable films less than 25 microns (1 mil) in thickness during the adhesive lamination process without introducing holes into the film. Thus, when adhesive lamination has been used to attempt to make composite sheets with thinner films, the composite sheets have not exhibited the fluid barrier properties (*e.g.*, hydrostatic head, dynamic fluid transmission) desirable for a composite sheet designed for use in absorbent articles or medical apparel.

Thermal lamination of moisture vapor permeable films less than 25 microns thick has similarly resulted in composite sheet materials with inadequate barrier properties. When composite sheets are made by thermally laminating a thin film to a fibrous substrate, the thin film handling problems associated with adhesive lamination as described above are encountered. In addition, to carry out a thermal lamination, the film must be subjected to elevated temperatures and pressures so as to soften the film and force it into mechanical

engagement with the fibrous substrate. Generally, the peel strength between the film and the fibrous substrate increases with increasing extrusion melt temperatures and increasing nip pressures. Unfortunately, when moisture vapor permeable films with a thickness of less than 25 microns are subjected to the elevated temperatures and pressures needed to obtain adequate peel strength in the composite sheet, small holes develop in the film such that the composite sheet does not exhibit the fluid barrier properties desired in a composite sheet for use in absorbent articles or medical apparel. These small holes can result from the non-uniform temperature throughout the web during bonding and from high bonding pressures used in the prior art.

Extrusion coating processes disclosed in the prior art are similarly unable to generate a composite sheet with a thin moisture vapor permeable film of less than 25 microns that also has the barrier properties and moisture vapor transmission properties desirable for use in medical apparel and absorbent article applications. In an extrusion coating process, the polymer that forms the film is melted at an elevated temperature to reduce its viscosity such that when the polymer melt is coated onto the fibrous substrate and passed through a nip, the melt is pressed into engagement with the fibrous network of the substrate. Unfortunately, the low viscosity of the melted polymer, the pressure of the nip, and a thinness of the film each contribute to the generation of small holes in the film. In addition, thinner films are more susceptible to fiber protrusion through the film which also contributes to small holes.

Accordingly, there is a need for a composite sheet material that acts as a barrier to fluids, yet is also highly permeable to moisture vapor. There is also a need for a sheet material that readily transmits moisture vapor, but significantly deters the passage of bacteria, viruses and odors associated with such fluids. There is a further need for such a moisture vapor permeable, fluid impermeable composite sheet material that is also durable, strong, and flexible enough to be used in apparel and absorbent articles, and can be produced in an economical fashion, i.e., film extrusion and lamination in one process. Specifically, there is a need for a composite sheet material with a moisture vapor permeable film that is less than 25 microns thick, exhibits excellent moisture vapor transmission, high peel strength, and barrier properties sufficient to prevent passage of liquids under static and dynamic loading conditions. Finally, there is a need for a process for producing such a composite sheet material.

### Summary of the Invention

The invention provides a moisture vapor permeable, substantially liquid impermeable composite sheet material comprising a fibrous substrate and a moisture vapor permeable thermoplastic film layer. The moisture vapor permeable film has an average thickness of less than 25 microns, a peel strength of at least 0.1 N/cm, a dynamic fluid transmission of less than about 0.75 g/m<sup>2</sup> when subjected to an impact energy of about 2400 joules/m<sup>2</sup>, a hydrostatic head of at least 60 cm, and a moisture vapor transmission rate, according to the desiccant method, of at least 2800 g/m<sup>2</sup>/24hr. The moisture vapor permeable film is preferably comprised of at least about 50% by weight of polymer selected from the group of block copolyether esters, block copolyether amides, polyurethanes, polyvinyl alcohol, and combinations thereof. Preferably, the moisture vapor permeable film has an average thickness of less than 20 microns and it exhibits a hydrohead of at least 120 cm, a dynamic fluid transmission of less than about 0.65 g/m<sup>2</sup> when subjected to an impact energy of about 2400 joules/m<sup>2</sup>, and it has a moisture vapor transmission rate, according to the dessicant method, of at least 3200 g/m<sup>2</sup>/24hr.

The preferred composite sheet is substantially free of micropores, and substantially no liquid moisture passes through the sheet when tested according to the liquid moisture seepage test. In addition, the preferred composite sheet, when tested according to ASTM F1671, prevents the passage of microbes with a diameter greater than 0.025 microns.

### Brief Description of the Drawings

The accompanying drawings, which are incorporated in and constitute a part of this specification, illustrate the presently preferred embodiments of the invention and, together with the description, serve to explain the principles of the invention.

Figure 1 is a cross-sectional view of the composite sheet structure of the invention.

Figure 2 is a cross-sectional view of a composite sheet structure according to an alternative embodiment of the invention.

Figure 3 is a schematic representation of a process by which the composite sheet structure of the invention can be made.

Figure 4 is a schematic representation of an alternative process by which the composite sheet structure of the invention can be made.

Figure 5 is a simplified illustration of an apparatus used for measuring dynamic fluid transmission of a sheet material.

Figure 6 is a simplified illustration of an apparatus used for measuring the moisture vapor transmission rate of a sheet material.

### Detailed Description of the Invention

Reference will now be made in detail to the presently preferred embodiments of the invention, examples of which are illustrated below.

The liquid impermeable, moisture vapor permeable composite sheet structure of the invention is shown in Figure 1. The composite sheet 10 is comprised of a fibrous substrate 14 to which a moisture vapor permeable and substantially liquid impermeable film 12 is adhered. Such composite sheets are sometimes referred to as laminate structures. The moisture vapor permeable film is substantially free of pinholes or pores, yet still has a relatively high rate of moisture vapor transmission. As used herein, "pinholes" means small holes inadvertently formed in a film either during manufacture or processing of the film, while "pores" means small holes in a film that are intentionally formed in the film in order to make the film porous to air, moisture vapor or liquids. In an alternative embodiment of the invention shown in Figure 2, the composite sheet structure may be comprised of a moisture vapor permeable film layer 12 with two fibrous substrates 14 and 16, each comprised of synthetic polymer fibers, adhered on opposite sides of the film layer.

In the preferred embodiment of the invention, the moisture vapor permeable, substantially liquid impermeable film is a polyether block copolymer such as copolymers comprised of block copolyether esters, block copolyether amides, polyurethanes, polyvinyl alcohols, or combinations thereof. The fibrous substrate 14 is preferably comprised of synthetic polymer fibers in a form to which the moisture vapor permeable film can be adhered. The substrate 14 may be a woven or nonwoven structure, but for cost reasons, nonwoven textile structures are preferred for most applications.

The fibrous substrates 14 and 16 should exhibit strength, permeability, and softness properties that are desired for the end use for which the composite sheet is to be applied. For example, where the composite sheet 10 is to be used in an absorbent article, the substrates 14 and/or 16 should preferably have a tensile strength of at least 1 N/cm and an elongation of at least 30% in both the machine and cross directions. The machine direction is the long direction within the plane of the sheet, i.e., the direction in which the sheet is produced. The cross direction is the direction within the plane of the sheet that is perpendicular to the machine direction. More preferably, the fibrous substrates have a tensile strength



of at least 1.5 N/cm and an elongation of at least 50% in both the machine and cross directions. Preferably, the fibrous substrate also has a porous structure that enhances both moisture permeability through the composite sheet and physical bonding between the film and substrate layers of the composite sheet.

5           One preferred nonwoven material for the fibrous substrates 14 and 16 is a fibrous polyolefin nonwoven web. Suitable polyolefin materials include polypropylene and polyethylene spunbonded webs, scrims, woven slit films, carded webs, flashspun webs, and woven or nonwoven sheets comprised of blends of polyolefin fibers or of polyolefin fibers and other fibers. Webs of  
10 polyolefin fibers can be made with a variety of desirable properties, including good vapor permeability, flexibility, softness and strength. A polyolefin sheet material that has been advantageously used for the fibrous substrate in the invention is TYPAR® spunbonded polypropylene sheet material. TYPAR® is a registered trademark of DuPont. Another fibrous polyolefin sheet material that  
15 has been advantageously used in the composite sheet of the invention is a carded, thermally-bonded polypropylene nonwoven material commercially available from Fiberweb of Simpsonville, South Carolina, under the trade designation HEC.

          Another preferred nonwoven material for the fibrous substrates 14 and 16 is a fibrous nonwoven web comprised of a blend of polyolefin and polyester  
20 fibers. Suitable polyolefin fibers include polypropylene and polyethylene staple fibers. Suitable polyester fibers include staple fibers made of polyethylene terephthalate (PET). Webs of polyolefin/polyester fiber blends can be made with a variety of desirable properties, including good vapor permeability, flexibility, softness and strength. One type of polyester fiber that has been blended with  
25 polyolefin fibers in the fibrous substrate are shaped polyester fibers with a scalloped-oval cross-section as disclosed in U.S. Patent 3,914,488 to Garrafa (assigned to DuPont), which is hereby incorporated by reference. It is believed that such shaped fibers create channels in the fibrous substrate through which moisture vapor can be more efficiently conveyed through the composite sheet.

30           Substrates 14 and 16 may alternatively be comprised of webs of other synthetic polymer materials such as polyamides, bicomponent fibers made of a polyolefin and one or more other polymers, or blends of polyolefin fibers and fibers comprised of other synthetic materials or other natural fibers such as cotton or cellulose fibers. The substrates 14 or 16 should have a side on which  
35 substantially few fibers extend out from the plane of the fibrous substrate, in other words, a fibrous substrate with a relatively smooth side. This smooth side of the substrate is critical when laminating a very thin film (< 25 microns) to the fibrous substrate. If the film is laminated to the surface of a fibrous substrate that is not

relatively smooth, fibers that protrude out from the plane of the substrate will likely protrude through the film, which will create pinholes and thereby allow liquid seepage.

Film layer 12 of the composite sheet structure 10 is a moisture vapor permeable and substantially liquid impermeable film. The film layer is preferably extruded and then laminated onto the fibrous substrate 14 in a single process. Film layer 12 comprises a thermoplastic polymer material that can be extruded as a thin, continuous, nonporous, substantially liquid impermeable, moisture vapor permeable film. Layer 12 is preferably comprised primarily of a block polyether copolymer, such as a polyether ester copolymer, a polyether amide copolymer, a polyurethane copolymer, polyvinyl alcohol, or a combination thereof. Preferred copolyether ester block copolymers for film layer 12 are segmented elastomers having soft polyether segments and hard polyester segments, as disclosed in U.S. Patent No. 4,739,012 (assigned to DuPont). Suitable polyether ester block copolymers are sold by DuPont under the name Hytrel®. Hytrel® is a registered trademark of DuPont. Suitable copolyether amide copolymers for film layer 12 are copolyamides available under the name Pebax® from Atochem Inc. of Glen Rock, New Jersey, USA. Pebax® is a registered trademark of Elf Atochem, S.A. of Paris, France. Suitable polyurethanes for use in film layer 12 are thermoplastic urethanes available under the name Estane® from The B.F. Goodrich Company of Cleveland, Ohio, USA.

The mixing of the thermoplastic polymer or blends of polymers that comprise the film layer of the sheet structure of the invention can be conducted according to methods and techniques known in the art, e.g., by physical tumble blending followed by extrusion and mixing in a single screw extruder equipped with a mixing head such as those available from Davis-Standard Corp. (Pawcatuck, Rhode Island, USA) or a twin screw compounding extruder such as those available from Warner-Pfliederer (Ramsey, New Jersey, USA) and Bersdorf Corporation (Charlotte, North Carolina, USA). Alternatively, loss in weight or volumetric feeders such as those available from K-Tron America (Pitman, New Jersey, USA) may be used to control the composition being fed to the extruders.

It has now been found that composite sheets with very thin moisture vapor permeable films (less than 25 microns in thickness) can be constructed in a manner such that the thin film is substantially free of pinholes. It has further been found that these moisture vapor permeable films can be attached to the fibrous substrate of the composite sheet in a way that the composite sheet retains a very high rate of moisture vapor transmission. In order to construct a composite sheet of a fibrous substrate and a very thin moisture vapor permeable film, it is

necessary that the film extrusion and lamination processes be conducted in a single step so as to make independent handling of the thin film unnecessary. In order to prevent the formation of pinholes during lamination, it has been found that the pressure applied to the film during the lamination process must be kept  
5 low in order to prevent the formation of non-uniform thin areas in the film where pinholes are likely to develop, especially due to impact related forces on the film.

Several methods have been found for making a composite sheet material with good peel strength between a thin moisture vapor permeable film and a fibrous substrate without the application of high pressure during the  
10 lamination process. According to one preferred process for making the composite sheet, an adhesive is applied to the surface of the fibrous substrate to which the moisture vapor permeable film is to be attached prior to application of the film. The adhesive is preferably applied to the substrate in a dispersed spray pattern at a basis weight of between 3.2 and 38.7 mg/cm<sup>2</sup> (0.5 and 6 mg/in<sup>2</sup>). It is important  
15 that the applied adhesive cover less than 75%, and more preferably less than 50%, and most preferably less than 25%, of the surface of the fibrous substrate so that the film layer coated over the adhesive will be discretely bonded to the fibrous substrate and the adhesive will not significantly reduce the moisture vapor transmission rate of the composite sheet. When an adhesive is used to help bond  
20 the film layer to the fibrous substrate of the composite sheet, it is preferred that the substrate be made of a material that is not compatible with the polymer of the moisture vapor permeable film. "Compatibility" of thermoplastic materials is an art-recognized term that refers, generally, to the degree to which the thermoplastic materials are miscible and/or interact with each other. Similarly, "incompatible"  
25 materials, as used herein, means polymer materials that are substantially immiscible or do not interact with each other. Incompatible materials do not wet each other well, nor do they adhere well to each other, even when heated. For example, when an adhesive is used to attach a copolyether ester elastomer film to a fibrous substrate, the preferred material for the fibrous substrate would be made  
30 of an incompatible polymer such as a polyolefin.

A preferred adhesive is a pressure sensitive hot melt adhesive such as a linear styrene isoprene styrene ("SIS") hotmelt adhesive, but it is anticipated that other adhesives, such as polyester or polyamide powdered adhesives, hotmelt adhesives with a compatibilizer such as polyester, polyamide or low residual  
35 monomer polyurethanes, other hotmelt adhesives, or other pressure sensitive adhesives could be utilized in making the composite sheet of the invention. Preferably the adhesive is applied to the surface of the fibrous sheet by an optional glue applicator 39, as shown in Figures 3 and 4, just before the polymer melt that

will form the moisture vapor permeable film layer is extruded onto the substrate. Applicator 39 may comprise a Series 6000 Melter and CF215 Applicator from the Nordson Corporation of Norcross Georgia. Alternatively, the adhesive may be applied to the fibrous substrate and then covered with a release paper and rolled up for storage and subsequent film lamination in another step. The moisture vapor permeable film can then be extrusion coated over the adhesive and bonded to the fibrous substrate as described in detail below. With this approach, it is believed that the heat from the film melt is sufficient to soften the adhesive in order to promote bonding.

10           According to another preferred process for making a composite sheet material with good peel strength between a thin moisture vapor permeable film and a fibrous substrate that does not require the application of high pressure during the lamination process, the film is extrusion coated directly onto the fibrous substrate without the introduction of a separate adhesive between the  
15           fibrous substrate and the film. In order to obtain good peel strength in the absence of an adhesive or the application of high pressure, the fibrous substrate is made with a blend of both fibers that are compatible with the polymer of the moisture vapor permeable film and fibers that are incompatible with the film. For example, if the film is a copolyether ester elastomer, the fibrous substrate preferably  
20           includes both compatible polyester fibers and incompatible polyolefin fibers. Good bonding can be achieved between a copolyether ester film and the polyester fibers of the fibrous substrate without the application of a high pressure bonding pressure to the film. However, it has been found that if the fibrous substrate includes too high of a percentage of highly compatible fibers, the film bonds to so  
25           many of the fibers of the substrate that the transmission of moisture vapor through the composite sheet material is reduced significantly. This reduction in the moisture vapor transmission rate is believed to be related to the film to fiber bond area because the bonded areas will have a significantly reduced moisture vapor transmission rate. Therefore, increasing the film to fiber bond area reduces the  
30           moisture vapor transmission rate. As illustrated in Examples 17-21 below, when a copolyether ester elastomer film is extrusion coated on various fibrous substrates, peel strength is improved when the substrate includes polyester fibers. Examples 30-41 illustrate that moisture vapor transmission goes down if the percentage of polyester fibers in the substrate gets too high.

35           In an extrusion coating process, a uniform molten extrudate is coated on the fibrous substrate. The molten polymer and the substrate are brought into more intimate contact as the molten polymer cools and bonds with the substrate. Such contact and bonding are normally enhanced by passing the layers through a

nip formed between two rolls. Alternatively, the molten polymer may be pulled into contact with the fibrous substrate by passing the coated substrate over a suction inlet such that the vacuum pulls the molten polymer into contact with the substrate as the polymer cools and bonds with the substrate. The bonding may be  
5 further enhanced by subjecting the surface of the substrate that is to contact the film to surface treatment, such as corona treatment, as is known in the art and described in Modern Plastics Encyclopedia Handbook, p. 236 (1994), which is hereby incorporated by reference.

One preferred means for applying the film layer 12 to the substrate 14  
10 is illustrated in Figure 3. As can be seen in Figure 3, the thermoplastic polymer is fed in pellet form, along with any additives, into the inlet 26 of the extruder hopper 24. The polymer is melted and mixed in the screw extruder 20 at a screw speed in the range of 10 to 200 rpm, depending on the dimensions of the extruder and the properties of the polymer. The melted mixture is discharged from the  
15 extruder under pressure through the heated line 28 to a flat film die 38. The polymer is discharged at a temperature above the melting temperature of the mixture, and preferably at a temperature in the range of 180° to 240° C. The polymer extrusion melt 40 discharging from the flat film die 38 coats the fibrous substrate 14. As discussed above, an adhesive applicator 39 may be used to apply  
20 an adhesive to the surface of the fibrous substrate 14 just before the substrate is coated with the extrusion melt 40.

Preferably, the substrate passes under the die at a speed that is coordinated with the speed of the extruder so as to obtain a very thin film thickness of less than 25 microns. The coated substrate enters a nip formed  
25 between the rolls 34 and 36, which rolls are maintained at a temperature selected to obtain a composite sheet with a desired peel strength and moisture vapor permeability. The temperature of the rolls 34 and 36 is within the range of 10° to 120° C. Higher roll temperatures have been found to yield a composite sheet with a higher peel strength, while lower roll temperatures have been found to yield  
30 composite sheets with a higher moisture vapor permeability. Preferably, roll 34 is a smooth rubber roller with a low-stick surface coating while the roll 36 is a metal roll. A textured embossing roll may be used in place of the metal roll for the roll 36 if a composite sheet with a more textured film layer (and higher surface area) is desired. Passing the coated substrate through the nip formed between cooled rolls  
35 34 and 36 quenches the polymer melt while at the same time compressing the polymer melt 40 into contact with the fibers of the fibrous substrate 14.

The pressure within the nip must be low such that thin spots or pinholes are not formed in the film as the substrate and film pass between the rolls

34 and 36. In the apparatus shown in Figure 3, pressure cylinders (not shown) are used to apply force to the rolls 34 and 36 which, in turn, generates pressure within the nip. In the apparatus shown in Figure 3, cylinder pressures of 552 kPa (80 psi) results in the application of a force of 172 N/linear cm along the length of the 20 inch long rolls; cylinder pressures of 414 kPa (60 psi) generate a force of 129 N/linear cm on the rolls; cylinder pressures of 276 kPa (40 psi) generate a force of 86 N/linear cm on the rolls; cylinder pressures of 138 kPa (20 psi) generate a force of 43 N/linear cm on the rolls; and cylinder pressures of 35 kPa (5 psi) generate a force of 11 N/linear cm on the rolls. When thin moisture vapor permeable films are to be bonded on the apparatus shown in Figure 3, it is preferred that a force of less than 50 N/linear cm be applied to the rolls.

When the polymer melt cools, it forms the film layer 12 of composite sheet 10, which composite sheet is collected on a collection roll 44. If a trilaminate product like that shown in Figure 2 is desired, an additional substrate material 16 may be in the same manner, be laid on the other side of the extruded polymer melt 40 as the polymer passes between rolls 34 and 36.

Alternatively, a vacuum process can be applied in order to lightly contact the polymer melt and the fibrous substrate material. The vacuum process is similar to conventional extrusion coating except that vacuum is used to bond the two substrates instead of nip rolls. The film is sucked onto the fibrous substrate by applying a vacuum force against the underside of the substrate. The vacuum process optimizes adhesion while also producing products with good loft and hand.

According to another embodiment of the invention, the film layer 12 may be a moisture vapor permeable, substantially liquid impermeable multiple layer film structure. Such a film may be coextruded with layers comprised of the one or more of the above described preferred moisture vapor permeable film materials described herein. Such multiple layer moisture permeable films are disclosed in U.S. Patent 4,725,481 (assigned to DuPont), which is hereby incorporated by reference. Multiple layer films are especially useful in the composite sheet of the invention where it is desirable for the film layer 12 to have different properties on its different sides. For example, a composite sheet can be made with a bicomponent film layer 12 having one side made of a moisture vapor permeable polymer material that thermally bonds well to the fibrous substrate 14 and an opposite side comprised of another moisture vapor permeable polymer that bonds well to materials to which the composite sheet is to be applied. It is anticipated that a moisture vapor permeable film of three or more co-extruded layers could be utilized, as disclosed in U.S. Patent 5,447,783 to Horn (assigned to

DuPont) for the film layer of the composite sheet of the invention in order to obtain an overall desired set of physical and aesthetic properties for the composite sheet.

An alternative apparatus for extrusion coating a multiple layer polymer melt onto a fibrous substrate is schematically illustrated in Figure 4. As can be seen in Figure 4, one thermoplastic polymer is fed in pellet form, along with any additives, into the inlet 26 of the extruder hopper 24, while another thermoplastic polymer is fed in pellet form, along with any additives, into the inlet 26' of the extruder hopper 24'. The polymer is melted and mixed in the screw extruders 20 and 20' at screw speeds in the range of 5 to 200 rpm, depending on the dimensions of the extruders and the properties of the polymer. The melted mixture is discharged from the extruder under pressure through heated lines to a melt combining block 34 where a multiple layer melt is formed that is extruded as a multiple layer film through the flat film die 38. The polymer is discharged from the film die 38 at a temperature above the melting temperature of the polymer mixture, and preferably at a temperature in the range of 180° to 240° C. The polymer melt 40 discharging from the flat film die 38 coats the fibrous substrate 14 provided from a supply roll 46. As discussed above, an adhesive applicator 39 may be used to apply an adhesive to the surface of the fibrous substrate 14 that is to be coated with the polymer melt 40.

Preferably, the fibrous substrate 14 in the apparatus and process illustrated in Figure 4 passes under the die 38 at a speed that is coordinated with the speed of the extruder so as to obtain a very thin film thickness of less than 25 microns. The coated substrate enters a nip formed between the rolls 52 and 54, which rolls are maintained at a temperature selected to obtain a composite sheet with a desired peel strength and moisture vapor permeability. Preferably, roll 52 is a smooth rubber roller with a low-stick surface coating while the roll 54 is a metal roll. Roll 52 may be cooled by a water bath 48. When the polymer melt cools, it forms the film layer 12 of composite sheet 10, which composite sheet is collected on a collection roll 60. Idler rolls 50 maintain tension on the fibrous substrate and the composite sheet throughout the composite sheet formation process.

According to another embodiment of the invention, a thin moisture vapor permeable film could be used in conjunction with a microporous film to form a laminate film structure. Such a structure overcomes a number of the drawbacks associated with microporous films, namely bacteria and liquid seepage and high moisture impact values, without sacrificing the relatively high MVTR values, often  $>3,000 \text{ g/m}^2/24 \text{ hr}$ , obtainable with some microporous films. The

moisture vapor permeable films of the composite sheet of the present invention can be made compatible with polyolefin nonwoven materials and can also be made compatible with current microporous film compositions, such as those of polyolefinic composition. The moisture vapor permeable film layer of the composite sheet of the present invention and a microporous film can be joined via adhesive lamination or by direct extrusion coating. The moisture vapor permeable film could be combined with a fibrous substrate in a fashion consistent with the present invention. This fibrous substrate and moisture vapor permeable substantially liquid impermeable film can be joined to a microporous film in a fashion consistent with the present invention, such that the nonwoven fibrous substrate will be bonded to the first side of the moisture vapor permeable, substantially liquid impermeable film layer and the microporous film will be laminated to the opposing side of the film layer.

The composite sheet 10 is especially useful as a component in disposable absorbent articles. As used herein, the term "absorbent article" refers to devices which absorb and contain body exudates, and, more specifically, refers to devices which are placed against or in proximity to the body of the wearer to absorb and contain the various exudates discharged from the body. Absorbent articles include disposable diapers, incontinence briefs, incontinence undergarments, incontinence pads, feminine hygiene garments, training pants, pull-on garments, and the like. The term "disposable" is used herein to describe absorbent articles which are not intended to be laundered or otherwise restored or reused as an absorbent article (i.e., they are intended to be discarded after a single use and, preferably, to be recycled, composted or otherwise disposed of in an environmentally compatible manner). Composite sheet 10 has physical properties that make the sheet especially useful as the outside "backsheet" of a disposable absorbent article, which properties include the composite sheet material's permeability to moisture vapor, its substantial impermeability to liquids, and its strength and durability. The ability of the composite sheet 10 to readily transmit moisture vapor means that hygiene products incorporating the composite sheet 10 as the product's backsheet material are comfortable to the wearer. The composite sheet's impermeability to fluids allows the sheet to completely contain bodily fluids even when the sheet is subjected to a dynamic impact of the type experienced when a baby or other person wearing a wet absorbent article sits down hard. The strength and durability of the composite sheet 10 permits the sheet to remain intact even after being stretched, rolled and pulled in the process of manufacturing an absorbent article.



It is believed that the moisture vapor transmission rate ("MVTR") of a composite sheet material used as the backsheet of an absorbent article is important in reducing humidity and temperature inside the absorbent article, thereby potentially reducing the incidence of heat rash and other skin problems associated with such environmental conditions. For example, in order to reduce rash inducing humidity and heat buildup within a disposable absorbent article, it has been found that at least a portion of the article's backsheet, and preferably the entire backsheet, should have a moisture vapor transmission rate of at least about 1500 g/m<sup>2</sup>/24 hr, as measured by the desiccant MVTR measurement method described in the examples below. The composite sheet material of the present invention is capable of delivering an MVTR, as measured by the desiccant method, of at least about 2800 g/m<sup>2</sup>/24 hr, and composite sheets according to the invention can deliver an MVTR greater than 4000 g/m<sup>2</sup>/24hr.

In the composite sheet of the present invention, moisture vapor transmission is enhanced because the moisture vapor permeable film layer 12 is extruded directly onto the nonwoven substrate 14. This direct extrusion improves moisture transmission for a number of reasons. First, direct extrusion makes it possible to make composite sheets with very thin film layers of less than 25 microns in thickness. These thin films are highly permeable to moisture vapor but they are still substantially impermeable to liquids. Second, because the pressure applied against the film layer 12 during the extrusion coating process is very low, the film layer can be made as thin as 7 microns without risk of pinholes. Thus, an extremely thin film can be bonded to the substrate without risking loss of liquid barrier properties. Third, because the film layer is discreetly bonded to the fibrous substrate, whether by adhesive lamination or by thermal lamination using a substrate fiber blend that includes compatible fibers, such that significant portions of the film layer are not bonded to the fibers of the fibrous substrate, moisture vapor transmission can be significantly increased.

The composite sheet of the present invention exhibits the important property that it is substantially impermeable to liquids under conditions that are normally associated with the use of absorbent articles and protective medical apparel. The liquid impermeability of the composite sheet 10 has been characterized according to a number of tests, including a liquid moisture seepage test, a dynamic barrier test, a hydrostatic head test, and a number of microbial barrier tests.

The liquid moisture seepage test visually demonstrates the substantial liquid impermeability of the composite sheet 10. As described in the example below, this test determines whether a solution of food dye, isopropyl alcohol and

water passes through a sheet material. As can be seen in Examples 17-20 below, the dye in alcohol solution did not pass through the composite sheet 10 of the present invention. On the other hand, when the same test was conducted on a sheet comprised of a microporous film laminated to a nonwoven substrate, dye solution seepage was apparent (Comparative Example 1).

The dynamic fluid impact test demonstrates the ability of the composite sheet 10 to resist liquid transmission. The dynamic fluid impact test described in the examples below is designed to mimic the energy per unit area that an infant imparts to a diaper backsheet when abruptly going from a standing to a sitting position. Suitable sheet materials for a diaper backsheet should exhibit substantially no dynamic fluid transmission (i.e., less than  $1 \text{ g/m}^2$ ) when subjected to an impact energy of about  $1000 \text{ joules/m}^2$ , as is the case for the composite sheet 14 of the invention. More preferably, diaper backsheets exhibit substantially no dynamic fluid transmission when subjected to an impact energy of  $2400 \text{ joules/m}^2$  or more. As reported in Examples 1-20 below, the composite sheet of the invention passed less than  $0.5 \text{ g/m}^2$  of water when subjected to an impact energy of about  $2400 \text{ joules/m}^2$ .

The ability of the composite sheet 10 to act as a barrier to liquids also prevents the passage of most odors, bacteria, or viruses through the sheet. When a microporous film was tested according to a bacteria flux test used for evaluating porous sterile packaging materials (ASTM F 1608-95) (Comparative Example 1), the material did not pass this test because bacteria was found to pass through the sheet. On the other hand, the composite sheet 10 of the invention, by being impermeable to air during a one hour air porosity test (See Gurley Hill porosity data in Examples 17-25), satisfies the microbial barrier requirement for impermeable sterile packaging materials, as set forth in ISO standard 11607, section 4.2.3.3. As can be seen in Example 17 below, the composite sheet 10 has also been shown to prevent the passage of viruses when tested according to ASTM F1671. ASTM F1671 measures the resistance of materials used in protective clothing to penetration of blood-borne viruses such as the Hepatitis B virus (HBV), the Hepatitis C virus (HCV), and the Human Immunodeficiency Virus (HIV) that causes Acquired Immune Deficiency Syndrome (AIDS). This method measures passage of the surrogate Phi-X174 bacteriophage, which is similar in size to the HCV virus and smaller than the HBV and HIV viruses, through a sheet material.

The strength and durability of composite sheet 10 makes this sheet especially suitable for absorbent article and apparel products. This strength and durability allow the composite sheet 10 to remain intact even after being stretched,

rolled, compressed and pulled during the process of manufacturing an absorbent article or apparel product. It is also important that the composite sheet be strong and durable enough to remain intact when stretched, pulled and wetted during wearing of an absorbent article or apparel product made using composite sheet 10 as the sheet material. The strength and durability of composite sheet 10 has been characterized in terms of (1) tensile strength, (2) the degree to which the sheet will stretch before breaking (known as "elongation"), and (3) the amount of force required to peel the moisture vapor permeable film from the fibrous substrate of the composite sheet (known as "peel strength" or "delamination strength").

Tensile strength is determined by measuring the tensile force required to rupture a sample of sheet material. Elongation is a measure of the amount that a sample of sheet material will stretch under tension before the sheet breaks. The elongation is the length just prior to break expressed as a percentage of the original sample length. Preferably, a composite sheet material that is to be used as the backsheet in an absorbent article has a tensile strength of at least 1 N/cm and an elongation of at least 30% in both the machine and cross directions. More preferably, if the composite sheet of the invention is to be used as the backsheet in an absorbent article, it should have a tensile strength of more than 1.5 N/cm and an elongation of at least 50% in both machine and cross directions. In the composite sheet of the present invention, the tensile properties and elongation properties of the composite sheet are largely dependent on the tensile and elongation properties of the fibrous substrate. A sheet material with the preferred tensile strength and elongation remains intact when wrapped around rollers at high speed during manufacture of absorbent articles. The elongation also makes the articles more comfortable to wearers because the articles have some give so as to be more conformable to a wearer's body shape because a sheet material with this elongation generally has some elasticity. As can be seen in Examples 17-20 below, the composite sheet 10 of the invention has a tensile strength of about 6 N/cm in the machine direction and about 1.4 N/cm in the cross direction, and an elongation of about 26% in the machine direction and about 55% in the cross direction. The preferred polyether block copolymer film of the invention provides a degree of elasticity to a composite sheet material that makes the sheet especially useful in an absorbent article.

Peel strength is a measure of the force required to delaminate the moisture permeable film from the fibrous substrate of a composite sheet. When the composite sheet 10 is used as a backsheet in a disposable absorbent article, such as a diaper, it is important that the composite sheet have a peel strength of at least 0.15 N/cm, and more preferably at least 0.20 N/cm, and most preferably at

least 0.50 N/cm, so that the sheet will not delaminate during manufacture of the article or during use. Such a peel strength is especially difficult to achieve when low pressure is applied at the nip to bond the film and the fibrous substrate because the composite exhibits reduced mechanical entanglement, and therefore  
5 reduced peel strength. Furthermore, adequate peel strength is even more difficult to achieve when the moisture vapor permeable film is chemically incompatible with the fibrous substrate, as is the case when a moisture permeable film comprised solely of a polyether ester block copolymer is coated on a polyolefin-based substrate. "Compatibility" of thermoplastic materials is an art-recognized  
10 term that refers, generally, to the degree to which the thermoplastic materials are miscible and/or interact with each other. Similarly, "incompatible" materials, as used herein, means polymer materials that are substantially immiscible or do not interact with each other. Incompatible materials do not wet each other well, nor do they adhere well to each other, even when heated.

15 The film layer in sheet structures according to the invention may additionally contain conventional additives, such as pigments and fillers (e.g.  $\text{TiO}_2$ , calcium carbonate, silicas, clay, talc) and stabilizers, such as antioxidants and ultraviolet absorbers. These additives are used for a variety of purposes, including reducing the cost of the film layer of the composite sheet structure, and  
20 altering the morphology of the film layer of the sheet structure. However, such additives have been found to reduce moisture vapor transmission through the sheet structure. It is important to maintain the amount of additive in the film at a level that does not result in a moisture vapor transmission rate for the sheet that falls outside of the range required for a particular application. The film layer may  
25 be comprised of between 0.01% and 30% of additive material, and more preferably between 0.5% and 7% of an inert filler material.

In terms of approaches to bond the composite sheet material to other components of an absorbent article, and more particularly to bond the moisture vapor permeable, liquid impermeable film layer of the composite sheet to other  
30 components, it has been observed that only certain methods of bonding will form bonds of sufficient strength to survive forces encountered in normal use, particularly after the film layer has been subjected to fluid contact and has absorbed fluid. Without wishing to be bound by theory, it is presently believed that the moisture vapor permeable film layers of interest in accordance with the  
35 present invention provide the desired superior performance properties in terms of moisture vapor transmission due to their comparatively high moisture content under in-use conditions. This comparatively high moisture content, however, is

presently believed to have negative implications on the bond strength of the bond between certain conventional hot melt adhesives and the film layer.

One approach which has proven satisfactory is to utilize a polyurethane-based adhesive in accordance with the conventional adhesive application techniques and equipment generally well known in the art, such as described above. Another approach, which is presently preferred, is to utilize the multiple layer, co-extruded film layer described above with reference to the aforementioned and incorporated U.S. Patent No. 4,725,481 to Ostapchenko, which disclosed a multiple layer film having a hydrophobic layer on the side of the film that was bonded to a nonwoven material and a hydrophilic layer on the opposite side of the film. Applicants have now found it beneficial to utilize a multiple layer moisture vapor permeable film wherein the multiple layer film structure (in a bi-layer execution) is extruded onto a fibrous substrate material with the comparatively more hydrophobic elastomer layer facing outwardly from the substrate and the comparatively more hydrophilic elastomer layer facing toward the substrate. For a given thickness, the hydrophobic elastomer layer typically exhibits a lower MVTR performance than the hydrophilic elastomer layer due to its comparatively lower moisture content under in-use conditions. However, when employed in a comparatively thin layer, the effect of the hydrophobic lower moisture content film layer does not significantly diminish the MVTR performance of the overall composite sheet.

Due to the comparatively low moisture content of the hydrophobic elastomer layer, conventional hot melt adhesives and bonding techniques may be utilized to successfully form bonds of adequate strength between the composite sheet and other components of the absorbent article even when the film has been wetted. Accordingly, by utilizing a co-extruded, multiple layer, multi-chemistry film layer, a composite sheet can be provided that exhibits both the desired performance properties for the composite sheet of the present invention and can be bonded to other components of absorbent articles via conventional adhesive bonding techniques. (See Examples 17-20 below.)

Quite unexpectedly, additional performance benefits have been discovered through the use of multiple layer films in composite sheets used in constructing absorbent articles. More particularly, the use of a multiple layer film comprising a three-layer structure with a hydrophobic elastomer layer on both facing surfaces surrounding a hydrophilic elastomer layer is believed to deliver improved tactile qualities when extruded onto a fibrous substrate to form a composite sheet. Again without wishing to be bound by theory, it is believed that the comparatively lower moisture content of the hydrophobic film layers results in a drier tactile

impression when the fibrous substrate layer is touched or palpated, particularly when the fibrous substrate layer is comparatively thin. Such a multiple layer (tri-layer) embodiment of a composite sheet material would therefore provide both an improved bondability with conventional adhesive techniques and an improved tactile impression from the side of the fibrous substrate layer. Optionally, as discussed above, truly dual-sided configurations could be constructed analogously to Figure 2 wherein the multiple layer/tri-layer film structure is faced on both sides with a fibrous substrate layer. Optionally, as discussed above, truly dual-sided configurations could be constructed analogously to Figure 2 wherein the multiple layer/tri-layer film structure is faced on both sides with a fibrous substrate material to provide an enhanced tactile impression from both sides. Such an execution is believed to be particularly desirable for such applications as leg cuffs, waistbands, side panels, and other aspects of absorbent articles such as diapers where a wearer may contact both opposing surfaces of the composite sheet material.

The following non-limiting examples are intended to illustrate the product and process of the invention and not to limit the invention in any manner.

#### EXAMPLES

In the description above and in the non-limiting examples that follow, the following test methods were employed to determine various reported characteristics and properties. ASTM refers to the American Society for Testing and Materials, TAPPI refers to the Technical Association of Pulp and Paper Industry, and ISO refers to the International Organization for Standardization.

Basis weight was determined by ASTM D-3776, which is hereby incorporated by reference, and is reported in g/m<sup>2</sup>.

Composite Sheet Thickness was determined by ASTM method D 1777-64, which is hereby incorporated by reference, and is reported in microns.

Film Thickness, is reported in microns, and was determined as follows:

$$\text{Film thickness} = \frac{(\text{composite sheet sample weight}) - (\text{substrate basis weight})(\text{sample area})}{(\text{sample area})(\text{density of film material})}$$

Tensile strength was determined by ASTM D 1682, Section 19, which is hereby incorporated by reference, with the following modifications. In the test a 2.54 cm by 20.32 cm (1 inch by 8 inch) sample was clamped at opposite ends of the sample. The clamps were attached 12.7 cm (5 in) from each other on the

sample. The sample was pulled steadily at a speed of 5.08 cm/min (2 in/min) until the sample broke. The force at break was recorded in Newtons/cm as the breaking tensile strength.

5        Elongation to Break of a sheet is a measure of the amount a sheet stretches prior to failure (breaking) in a strip tensile test. A 1.0 inch (2.54 cm) wide sample is mounted in the clamps - set 5.0 inches (12.7 cm) apart - of a constant rate of extension tensile testing machine such as an Instron table model tester. A continuously increasing load is applied to the sample at a crosshead speed of 2.0 in/min (5.08 cm/min) until failure. The measurement is given in percentage of stretch prior to failure. The test generally follows ASTM D1682-  
10       64.

Peel strength is measured according to a test that generally follows the method of ASTM D882-83, which is hereby incorporated by reference. The test was performed used a constant rate of extension tensile testing machine such as an  
15       Instron table model tester. A 2.54 cm (1.0 in) by 20.32 cm (8.0 in) sample is delaminated approximately 3.18 cm (1.25 in) by initiating a separation between the fibrous substrate and the moisture vapor permeable film. The separated sample faces are mounted in the clamps of the tester which are set 5.08 cm (2.0 in) apart. The tester is started and run at a cross-head speed of 50.8 cm/min (20.0  
20       in/min). The computer starts picking up readings after the slack is removed, nominally a 5 gram pre-load. The sample is delaminated for about 12.7 cm (5 in) during which sufficient readings are taken to provide a representative average of the data. The peak load and average peel strength is given in N/cm. For samples that are peeled the entire 5 inches the average peel strength is considered to be the  
25       peel strength. For samples that do not peel the entire 5 inches due to either full bond conditions or failures in the substrates, the peak load is considered to be the peel strength.

Bond Strength is a measure of the adhesive bond strength between a composite sheet and a 1.2 mil polyethylene film. This is also defined as the  
30       strength of the adhesive bond used to construct absorbent articles wherein an adhesive is used to join other materials to the composite structure. Samples were prepared by applying a conventional hotmelt adhesive useful in absorbent article construction to the film side of the composite structure. The adhesive can be any adhesive useful in constructing absorbent articles, one such adhesive useful in  
35       article construction is designated as H2031 available from AtoFindley Adhesives, Inc. 11320 Watertown Plank Road, Wauwatosa, WI 53226-3413. This adhesive is a pressure sensitive linear SIS, styrene-isoprene-styrene, hotmelt adhesive. The adhesive is applied at a glue weight of .009 g/in<sup>2</sup> using a Meltex EP34S spray

application head. The temperature of the adhesive at the nozzle is 330 F. This spray application head is available from the Nordson Corporation, 2905 Pacific Drive, Norcross, GA 30071-1809. Once the glue is applied, a polyethylene film, 1.2 mil thickness, is adhered to the composite structure by placing the

5 polyethylene in contact with the adhesively coated composite structure and applying pressure by rolling the sample with a hand roller similar to those used for wallpaper. Samples are then cut to a 2.54 cm (1 inch) width and 10.16 cm (4 inch) length with 1 inch of the length being the adhesively bonded area. At least 3 samples are prepared for both wet and dry bond. For the wet bond the adhesive

10 end of the sample is placed in a petri dish filled with distilled water for 15 minutes. Immediately upon removal from the water the sample is tested as follows. Dry bond testing requires only the sample prep described previously and then testing as follows. The free, unbonded, ends of the sample are mounted in the clamps of the tester which are set 5.08 cm (2.0 in) apart. The tester is started

15 and run at a cross-head speed of 50.8 cm/min (20.0 in/min). The computer starts picking up readings after the slack is removed, nominally a 5 gram pre-load. The sample is peeled completely, about 2.54 cm (1 inch) during which sufficient readings are taken to provide a representative average of the data. The peak load and average bond strength is given in N/cm. For samples that are peeled the entire

20 1 inch, the average bond strength is considered to be the bond strength. For samples that do not peel the entire 1 inch due to either bond conditions or failures in the substrates, the peak load is considered to be the bond strength.

Water Absorption is measured according to ASTM D570, which is hereby incorporated by reference.

25 Moisture Vapor Transmission Rate (MVTR) was determined by a method that is based in part on ASTM E96, which is hereby incorporated by reference, and is reported in  $\text{g/m}^2/24 \text{ hrs}$ .

This method is referred to as the "dessicant method" for measuring moisture vapor transmission rate as set forth below. Briefly summarizing this

30 method, a defined amount of desiccant ( $\text{CaCl}_2$ ) is put into a flanged "cup" like container, see Fig 6 shown with a partial cutaway. The sample 155 material is placed on the top of the container 157 and held securely by a retaining ring 152 and gasket 153. The assembly is then weighed and recorded as the initial weight. The assembly is placed in a constant temperature ( $40^\circ\text{C} \pm 3^\circ\text{C}$ ) and humidity

35 ( $75\% \text{ RH} \pm 3\%$ ) chamber for five (5) hours. The assembly is then removed from the chamber, sealed to prevent further moisture intake, and allowed to equilibrate for at least 30 minutes at the temperature of the room where the balance is located. The amount of moisture absorbed by the  $\text{CaCl}_2$  156 is determined gravimetrically



and used to estimate the moisture vapor transmission rate (MVTR) of the sample by weighing the assembly deducting the initial weight from the final assembly weight. The moisture vapor transmission rate (MVTR) is calculated and expressed in  $\text{g/m}^2/24 \text{ hr.}$  using the formula below. Samples are assayed in  
5 triplicate. The reported MVTR is the average of the triplicate analyses, rounded to the nearest 100. The significance of differences in MVTR values found for different samples can be estimated based on the standard deviation of the triplicate assays for each sample.

Suitable Analytical Balances for performing the gravimetric  
10 measurements include a Mettler AE240 or equivalent (300 g capacity) or a Sartorius 2254S0002 or equivalent (1000 g capacity). A suitable sample holding assembly comprises a cup 157 and retaining ring 152 machined from Delrin® (such as that available from McMaster-Carr Catalog #8572K34) with a gasket 153 made of GC Septum Material (Alltech catalog #6528). The dimensions of the  
15 cup, retaining ring and gasket are as follows: the dimensions of the cup are A which corresponds to the retaining ring outer diameter and cup flange diameter is 63mm, B is 55mm, C which is the retaining ring thickness is 5 mm, D which is the flange thickness is 6 mm, E is the cup height and the dimension is 55mm, F corresponds to the inner diameter of the cup and also the diameter of the opening  
20 in the retaining ring this dimension is 30 mm, G is the outer diameter of the cup which is 45mm. The dessicant comprises  $\text{CaCl}_2$  156 for U-tubes, available from Wako Pure Chemical Industries, Ltd., Richmond, VA. Product # 030-00525. The plastic food wrap comprises Saran Wrap, available from Dow Chemical Company, or equivalent. A suitable environmental chamber is available from  
25 Electro-Tech Systems, Inc, ETS, model 506A or equivalent. The temperature controller is ETS model 513A or equivalent, the humidity controller is ETS model 514 or equivalent, the heating unit is a Marley Electric Heating Model 2512WC (400 watts) or equivalent, the humidifier is ETS model 5612B or equivalent.

The  $\text{CaCl}_2$  can be used directly from a sealed bottle as long as the size  
30 of the lumps is such that they do not pass through a No. 10 sieve. Usually the top two-thirds of the bottle does not have to be sieved. However, the bottom third contains fines that should be removed by sieving. The  $\text{CaCl}_2$  can be used from a closed container without drying. It can be dried at  $200^\circ\text{C}$  for 4 hours if required.

Representative samples should be obtained from the materials to be  
35 tested. Ideally, these samples should be taken from different areas of the material so as to represent any variations present. Three samples of each material are needed for this analysis.

Samples should be cut into rectangular pieces approximately 1.5" x 2.5". If the samples are not uniform, clearly mark the area for which breathability is to be evaluated. If the samples are not bidirectional, clearly mark the side that is to be exposed to high humidity. For samples used in diapers and catamenials, this is usually the side that contacts the absorbent layer of the article or the wearer in the case of garments.

To begin a test session, (1) weigh approximately 15 grams of  $\text{CaCl}_2$  156 and place in the MVTR cup 157. Gently tap the cup 157 10 times on the bench top to distribute and lightly pack the  $\text{CaCl}_2$ . The  $\text{CaCl}_2$  156 should be level and about 1 cm from the top of the cup 157. Adjust the amount of  $\text{CaCl}_2$  until the 1 cm distance is achieved. Then (2) place the sample 155, with the high humidity side up (if required), over the opening in the top of the cup 157. Make sure that the sample overlaps the opening so that a good seal will be obtained. Next, (3) place the gasket material 153 and the retaining ring 152 on the top of the cup, aligning the screw holes and checking to make sure that the sample has not moved. Tighten the screws 154 to securely fasten the retaining ring 152 and seal the sample to the top of the cup. Care should be taken to not over tighten the screws 154 as this leads to distortion of some samples. If distortion of the sample occurs, loosen the screws 154 and tighten again. Then (4) weigh the MVTR cup assembled in step 3. Record this weight as the initial weight. This process should be conducted in a relatively short time per cup, <2 minutes.

After weighing the assembly, (5) place the sample in the CT/CH chamber for 5.0 hours (to the nearest minute). When the time has elapsed, (6) remove the sample from the CT/CH chamber, tightly cover it with plastic wrap secured by a rubber band. Record the time of sample removal to within the nearest minute. Allow samples to equilibrate for at least 30 minutes at the temperature of the room where the balance is located. After equilibration, (7) remove the plastic wrap and rubber band and weigh the cup. Record this weight as the final weight.

The MVTR is then calculated in units of  $\text{g H}_2\text{O}/24 \text{ hr}/\text{m}^2$  using the formula:

$$\text{MVTR} = \frac{(\text{final weight} - \text{initial weight}) \times 24.0}{\text{area of sample in meters} \times 5.0 (\text{time in chamber})}$$

where: 24.0 is used to convert the data to the 24 hour basis;  
the area of sample is equal to the open area of the mouth of the cup;  
and  
5.0 is the duration of the test in hours

Calculate the average MVTR for each set of triplicate. Round the average MVTR for each sample set to the nearest 100. Report this value as the MVTR for the sample of material.

5            Dynamic Fluid Transmission is measured with the apparatus 100 shown in Figure 7. According to this test, an absorption material 102 weighed to the nearest 0.0001 gram is placed directly on top of the energy absorbing impact pad 103. The absorption material 102 may comprise a No. 2 filter paper available from Whatman Laboratory Division, Distributed by VWR Scientific of Cleveland,  
10 OH. The absorption material should be able to absorb and retain the distilled water which passes through the sheet material being tested. The energy absorbing impact pad 103 is a carbon black filled cross linked rubber foam. The 5 inch by 5 inch square impact pad has a density of 0.1132 g/cm<sup>3</sup> and a thickness of 0.3125 inches. The impact pad 103 has a Durometer Value of A/30/15 according to  
15 ASTM 2240-91. A circular absorbent core material 104 measuring 0.0572 meters (2.25 inches) in diameter is weighed. The absorbent core material may comprise individualized, crosslinked wood pulp cellulosic fibers as described in U.S. Pat. No. 5,137,537 issued to Herron et al. on Aug. 11, 1992. The absorbent core material should be able to hold a sufficient amount of distilled water, e.g., at least  
20 about ten times its dry weight. The absorbent core has a basis weight of about 228 g/m<sup>2</sup>. The absorbent core material is then loaded with distilled water to about ten (10) times its dry weight.

A section of the backsheet material 105 to be tested is placed face down with the outside surface on a clean and dry tabletop. The loaded core  
25 material 104 is placed directly in the center of the backsheet material 105. The backsheet/core arrangement is then secured to the impact portion 107 of the impact arm 108 with a rubber band 109. The backsheet/core arrangement is positioned such that the core 104 is adjacent the bottom surface 110 of the impact portion 107. The impact arm 108 is raised to a desired impact angle to provide the  
30 desired impact energy. The impact arm 108 is dropped and the impact arm 108 is then immediately (about 1 second after impact) raised and the filter paper 102 is removed and placed on a digital scale. The mass of the wet filter paper is then recorded at the one minute mark. The dynamic fluid transmission value (DFTV) is calculated and expressed in g/m<sup>2</sup> using the following formula:

35

$$\text{DFTV} = \frac{\text{mass of the wet filter paper (grams)} - \text{mass of the dry filter paper (grams)}}{\text{impact area (m}^2\text{)}}$$

The impact area, expressed in  $m^2$ , is the area of the bottom surface 110 of the impact portion 107. The impact area is  $0.00317 m^2$ . The absorbent core material 104 should have an area slightly larger than that of the impact area of the surface.

Gurley Hill Porosity is a measure of the barrier strength of the sheet material for gaseous materials. In particular, it is a measure of how long it takes for a volume of gas to pass through an area of material wherein a certain pressure gradient exists. Gurley-Hill porosity is measured in accordance with TAPPI T-460 om-88 using a Lorentzen & Wettre Model 121D Densometer. This test measures the time of which 100 cubic centimeters of air is pushed through a one inch diameter sample under a pressure of approximately 4.9 inches of water. The result is expressed in seconds and is usually referred to as Gurley Seconds.

Bacterial Barrier for Sterile Packaging is measured according to ISO 11607 which states under section 4.2.3.2 that a material that is impermeable to air for one hour (according to an air porosity test) satisfies the standard's microbial barrier requirements. With regard to porous materials, section 4.2.3.3 of ISO 11607 states that there is no universally applicable method of demonstrating microbial barrier properties in porous materials, but notes that the microbial barrier properties of porous materials is typically conducted by challenging samples with an aerosol of bacterial spores or particulates under a set of test conditions which specify the flowrate through the material, microbial challenge to the sample, and duration of the test. One such recognized test is ASTM F 1608-95.

Viral Barrier properties were also measured according to ASTM F1671, which is hereby incorporated by reference. ASTM F1671 is a standard test method for measuring the resistance of materials used in protective clothing to penetration by blood-borne pathogens. According to this method, three samples of a sheet material being tested are challenged with  $10^8$  Phi-X174 bacteriophage, similar in size to the Hepatitis C virus (0.028 microns) and with a surface tension adjusted to 0.042 N/m, at a pressure differential of 2 psi (13.8 kPa) for a 24 hour period. Penetration of the sample by viable viruses is determined using an assay procedure. The test results are reported in units of Plaque Forming Units per milliliter PFU/ml. A sample fails if any viral penetration is detected through any of the samples. A positive and negative control is run with each sample set. The positive control was a microporous membrane with a pore size of 0.04 microns which passed 600 PFU/ml. The negative control was a sheet of Mylar® film, which passed 0 PFU/ml.

Liquid Moisture Seepage is detected using a solution of 70 parts isopropyl alcohol, 30 parts water and 1 part red dye food color. According to this

test, a sheet of a white absorbent blotting material measuring about 89 cm by 61 cm (35 in by 24 in) is placed on a flat surface and covered with a test sample of the same dimensions with the substrate side of the sample facing up. A 250 ml portion of the solution is poured on top of the test sample and covered with a  
5 template measuring about  $46 \frac{3}{4}$  cm by  $46 \frac{3}{4}$  cm (18 in by 18 in). A 4.5 kg (10 lb) weight is placed on top of the template for 10 minutes after which the weight, template and test sample are removed from the white blotting paper. The paper is then inspected for ink spots to determine whether seepage occurred.

10 Film Components

The film compositions described in the examples below were prepared by dry blending one or more copolyether ester thermoplastic elastomers and titanium dioxide. The individual components in the film compositions were as follows:

15 Hytrel® 3548 is a copolyether ester thermoplastic elastomer sold by DuPont, and having a melting point of 156° C, a vicat softening temperature of 77° C, a shore hardness of 35D, and a water absorption of 5%.

Hytrel® 4778 is a copolyether ester thermoplastic elastomer sold by DuPont, and having a melting point of 208° C, a vicat softening temperature of  
20 175° C, a shore hardness of 47D, and a water absorption of 2.3%.

Hytrel® 8206 is a copolyether ester thermoplastic elastomer sold by DuPont, and having a melting point of 200° C, a vicat softening temperature of 151° C, a shore hardness of 45D, and a water absorption of 30%.

Hytrel® 8171 is a copolyether ester thermoplastic elastomer sold by  
25 DuPont, and having a melting point of 150° C, a vicat softening temperature of 76° C, a shore hardness of 32D, and a water absorption of 54%.

TiO<sub>2</sub> Concentrate was a concentrate of 60% by weight particulate titanium dioxide pigment in high density polyethylene.

30 EXAMPLES 1 - 5

A copolyether ester film was adhesively laminated to a carded polypropylene nonwoven sheet with a basis weight of 27 g/m<sup>2</sup> (0.8 oz/yd<sup>2</sup>) obtained for Fiberweb North America Inc. of Simpsonville, South Carolina. The nonwoven sheet was comprised of carded polypropylene staple fibers, with fiber  
35 lengths generally ranging between 2.5 cm and 7.5 cm, that were thermally bonded. The polypropylene fiber carded sheet had a tensile strength of 8.3 N/cm (4.73 lb/in) in the machine direction and 1.5 N/cm (0.86 lb/in) in the cross

direction, and an elongation of 73% in the machine direction and 95% in the cross direction.

A pressure sensitive linear SIS hotmelt adhesive (H2031 from Ato Findley Adhesives, Inc. of Wauwatosa, Wisconsin) was applied to the nonwoven using a Series 6000 Melter and CF215 Applicator from the Nordson Corporation of Norcross Georgia. The adhesive was applied in a substantially continuous filament in a dispersed spiral spray pattern that was 12 inches wide using 15 applicator modules that were 0.875 inches on center. The individual spiral patterns were applied edge to edge with no substantial overlap of the spiral patterns from the various applicator modules. The line speed of the nonwoven during adhesive application was 400 fpm (122 m/min). The basis weight of the applied adhesive was 2 mg/in<sup>2</sup> (3 g/m<sup>2</sup>). The adhesive was covered with a release paper and the nonwoven coated with the adhesive was rolled up.

The nonwoven to which the adhesive and release paper had been applied was unrolled, the release paper was removed, and the adhesive coated side of the nonwoven was laminated with a polymer film comprised of 48% Hytrel® 8206 copolyether elastomer, 48% Hytrel® 8171 copolyether elastomer, and 4% titanium dioxide. The copolyether ester polymer was fed in pellet form into a 38mm diameter screw extruder that was connected to a heated die. The polymer was melted and then fed to a 36 cm by 250 micron die opening in the heated die block. The polymer melt was extruded from the die opening and was coated on the polypropylene nonwoven sheet over the applied adhesive as described above with regard to Figure 3. The film was joined to the fibrous nonwoven sheet in a nip that was spaced about 10 cm from the die opening. The nip was formed between a metal roll that faced the fibrous sheet and a rubber roll that faced the polymer melt.

In Examples 1-5, the line speed of the nonwoven was maintained at a constant 12 m/min (40 ft/min) and the film was extruded at constant rate (extruder speed of 12 rpm) in order to keep the thickness of the copolyether ester film constant. The polypropylene fiber sheet, the adhesive and the film were passed through the nip where the pressures on the nip were adjusted to form a variety of composite sheet structures. With the nip arrangement used in Examples 1-5, as shown in Figure 3, cylinder pressures of 552 kPa (80 psi) correspond to a force of 172 N/linear cm along the length of the 20 inch long rolls; cylinder pressures of 414 kPa (60 psi) generate a force of 129 N/linear cm on the rolls; cylinder pressures of 276 kPa (40 psi) generate a force of 86 N/linear cm on the rolls; cylinder pressures of 138 kPa (20 psi) generate a force of 43 N/linear cm on the

rolls; and cylinder pressures of 35 kPa (5 psi) generate a force of 11 N/linear cm on the rolls.

Other process conditions used in each example and the properties of the resulting composite sheets are set forth in Table 1 below.

5

Table 1

<u>EXAMPLE NUMBER.</u>	1 (90J4)	2 (90J3)	3 (90J2)	4 (90J5)	5 (90J6)
<u>Process Conditions</u>					
Adhesive temperature (°C)	ambient	ambient	ambient	ambient	ambient
Adhesive application density (g/m <sup>2</sup> )	3	3	3	3	3
Adhesive pattern	spiral	spiral	spiral	spiral	spiral
Line speed (m/min)	12	12	12	12	12
Extruded melt temp (°C)	220	220	220	220	220
Extruder speed 1 (RPM)	12	12	12	12	12
Die temperature (°C)	220	220	220	220	220
Cylinder pressure (kPa)	35	70	140	280	560
<u>Composite Properties</u>					
Film Thickness (microns)	25	25	25	24	24
MVTR -(Desiccant Method) (g/m <sup>2</sup> /day)	2900	3200	2900	2800	3000
Dynamic Impact (g/m <sup>2</sup> @ 2400 J/m <sup>2</sup> )	0.36	0.44	0.44	0.25	0.35
Peel Strength (N/cm)					
MD	0.95	1.04	full	1.03	full
CD	0.80	0.84	full	1.01	full

Examples 1-5 demonstrate that excellent peel strength can be obtained in a composite sheet at relatively low bonding pressures (Examples 1 and 2) when an adhesive is added between the film and the nonwoven. These examples also show that good moisture impact barrier properties can be obtained from a composite sheet with a thin moisture vapor permeable film when bonding pressures are kept low. Finally, these Examples support the belief that the film melt temperature is adequate to promote outstanding peel strength.

## EXAMPLES 6-10

A copolyether ester film was adhesively laminated to the fibrous polypropylene nonwoven sheet of Examples 1-5. First, an adhesive was applied to the moving nonwoven sheet at three different adhesive basis weights using the adhesive coating process as described with respect to Examples 1-5 above. The adhesive was covered with a release paper and the nonwoven coated with the adhesive was rolled up.

The nonwoven to which the adhesive and release paper had been applied was unrolled, the release paper was removed, and the adhesive coated side of the nonwoven was laminated with a polymer film comprised of 48% Hytrel® 8206 copolyether elastomer, 48% Hytrel® 8171 copolyether elastomer, and 4% titanium dioxide. The copolyether ester polymer was fed in pellet form into a 38 mm diameter screw extruder that was connected to a heated die. The polymer was melted and then fed to a 36 cm by 250 micron die opening in the heated die block. The polymer melt was extruded from the die opening and was coated on the polypropylene nonwoven sheet over the applied adhesive as described above with regard to Figure 3. The film was joined to the fibrous nonwoven sheet in a nip that was spaced about 10 cm from the die opening. The nip was formed between a metal roll that faced the fibrous sheet and a rubber roll that faced the polymer melt.

In Examples 6-10, the line speed of the nonwoven and the speed at which the film was extruded were varied to produce films of different thicknesses. The nip arrangement was like that of Examples 1-5, and the pressure cylinders were maintained at a constant pressure of about 140 kPa (20 psi). The process conditions used in each example and the properties of the resulting composite sheets are set forth in Table 2 below.



Table 2EXAMPLE NUMBER.

6	7	8	9	10
(90J7)	(90J8)	(90J9)	(90J2)	(90J1)

Process Conditions

Adhesive temperature (°C)	Ambient	Ambient	Ambient	Ambient	Ambient
Adhesive application density (g/m <sup>2</sup> )	3	3	3	3	3
Adhesive pattern	Spiral	Spiral	Spiral	Spiral	Spiral
Line speed (m/min)	12	17	17	12	9
Extruded melt temp (°C)	220	220	220	220	220
Extruder speed 1 (RPM)	12	12	6	12	16
Die temperature (°C)	220	220	220	220	220
Cylinder pressure (kPa)	140	140	140	140	140

Composite Properties

Film Thickness (microns)	17	12	7	25	37
MVTR -(Desiccant Method) (g/m <sup>2</sup> /day)	3300	3800	4800	2900	2600
Dynamic Impact (g/m <sup>2</sup> @ 2400 J/m <sup>2</sup> )	0.25	0.33	0.33	0.44	0.25
Peel Strength (N/cm)					
MD	full	full	full	full	1.15
CD	full	full	full	full	0.97

Examples 6-10 demonstrate that the MVTR of the composite sheet can be improved by as much as 85% by reducing film thickness from 25 microns (Ex. 9) to 7 microns (Ex. 8). These Examples further show that this improved MVTR can be obtained without a significant loss in peel strength or dynamic impact barrier properties. These Examples support the belief that adhesive application prior to film extrusion is a viable process, and when combined with low bonding pressures, results in composites with films as thin as 7 microns that have adequate peel strength and dynamic impact barrier properties. These Examples also illustrate that application of the adhesive at the relatively low basis weight of about 3 g/m<sup>2</sup> is adequate to provide good peel strength.

EXAMPLES 11- 16

A copolyether ester film was adhesively laminated to the fibrous polypropylene nonwoven sheet of Examples 1-5. First, an adhesive was applied to the moving nonwoven sheet at a coating density of 2 mg/in<sup>2</sup> (3 mg/cm<sup>2</sup>) as

described with respect to Examples 1-5 above. The adhesive was covered with a release paper and the nonwoven coated with the adhesive was rolled up.

The nonwoven to which the adhesive and release paper had been applied was unrolled, the release paper was removed, and the adhesive coated side  
5 of the nonwoven was laminated with a polymer film comprised of 48% Hytrel® 8206 copolyether elastomer, 48% Hytrel® 8171 copolyether elastomer, and 4% titanium dioxide. The copolyether ester polymer was fed in pellet form into a 38mm diameter screw extruder that was connected to a heated die. The polymer was melted and then fed to a 36 cm by 250 micron die opening in the heated die  
10 block. The polymer melt was extruded from the die opening and was coated on the polypropylene nonwoven sheet over the applied adhesive as described above with regard to Figure 3. The film was joined to the fibrous nonwoven sheet in a nip that was spaced about 10 cm from the die opening. The nip arrangement was like that of Examples 1-5, and the pressure cylinders were maintained at a  
15 constant pressure of about 140 kPa (20 psi).

In Examples 11-13, the adhesive coating density was varied while other process conditions were kept constant. In Examples 14-18, the adhesive coating density was varied while a second set of process conditions were maintained. The process conditions used in each example and the properties of  
20 the resulting composite sheets are set forth in Table 3 below.

Table 3

EXAMPLE NUMBER.

11 (90L2)	12 (90J2)	13 (90K2)	14 (90L4)	15 (90J7)	16 (90K3)
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Process Conditions

Adhesive temperature (°C)	Ambient	Ambient	Ambient	Ambient	Ambient	Ambient
Adhesive application density (g/m <sup>2</sup> )	1.6	3	6.2	1.6	3	6.2
Adhesive pattern	Spiral	Spiral	Spiral	Spiral	Spiral	Spiral
Line speed (m/min)	12	12	12	12	12	12
Extruded melt temp (°C)	220	220	220	220	220	220
Extruder speed 1 (RPM)	16	16	16	12	12	12
Die temperature (°C)	220	220	220	220	220	220
Cylinder pressure (kPa)	140	140	140	140	140	140

Composite Properties

Film Thickness (microns)	26	25	30	23	17	24
MVTR -(Desiccant Method) (g/m <sup>2</sup> /day)	2800	2900	2400	3000	3300	3000
Dynamic Impact (g/m <sup>2</sup> @ 2400 J/m <sup>2</sup> )	0.03	0.44	0	0.13	0.25	0.11
Peel Strength (N/cm)						
MD	0.59	full	full	0.49	full	full
CD	0.49	full	full	0.30	full	full

Examples 11-16 demonstrate that good peel strength can be obtained even at low adhesive basis weights (Exs. 11 and 14). These Examples also show that the open spiral pattern in which the adhesive is applied permits the application of relatively high adhesive basis weights without causing a substantial reduction in moisture vapor transmission rates (Exs. 13 and 16). This data also suggests that the film melt temperature is high enough to soften the adhesive to promote good bonding.

## EXAMPLES 17-21

A bi-layer copolyether ester polymer film was laminated to a nonwoven sheet comprised either of a polyester and polypropylene staple fiber blend or of 100% polyethylene staple fibers. No additional adhesive was used.

The nonwoven sheet material of Examples 17-20 was a 50/50 blend of polyester staple fiber (Dacron® Type 54 polyester fiber manufactured by DuPont) and polypropylene staple fibers (Danaklon Hy Comfort polypropylene fiber manufactured by Danaklon America, Inc. of Athens, Georgia). The

polyester and polypropylene staple fibers had fiber lengths of about 40 mm and a denier of 2. The fibers were carded and thermobonded at 143° C with a nip pressure of 40 daN/cm using a Kuester calender Hot-S-Roll. The nonwoven blend used in Examples 17-20 had basis weights ranging from 9.9 g/m<sup>2</sup> to 28.3 g/m<sup>2</sup>.

5           The nonwoven sheet material of Example 21 was comprised solely of spunbonded polyethylene fiber with a basis weight 28.3 g/m<sup>2</sup> manufactured by Polybond Company of Waynesboro, Virginia.

10           The bi-layer copolyether ester film used in Examples 17-21 had a Film Layer 1 comprised of 100% Hytrel® 4778 that made up 17%, by weight of the film, and a Film Layer 2 comprised of a blend of 48% Hytrel® 8171, 46% Hytrel® 8206 and 6% TiO<sub>2</sub> that made up 83% of the film (percents are by weight). The components for Film Layer 1 were mixed and fed in pellet form into a 4 inch (10.2 cm) inch diameter screw extruder that was connected to a melt combining block. The components for Film Layer 2 were also mixed and fed in  
15           pellet form into a different 3 inch (7.6 cm) inch diameter screw extruder that was connected to the same melt combining block. The components for Film Layers 1 and 2 were each melted and extruded to the melt combining block. The two layer melt was then fed to a 762 microns by 102 cm die opening in a heated die block. A bicomponent film with Film Layer 1 and Film Layer 2 was extruded from the  
20           die opening and was coated on the polypropylene nonwoven sheet as shown in Figure 4. The nonwoven sheet was spaced about 23 cm (9 in) below the opening of the die.

25           The nonwoven fiber sheet and the film were passed through a pair of nip rolls, as shown in Figure 4, to form the composite sheet structure. The nip rolls were subjected to air cylinder pressures of 207 kPa (30 psi) and the rolls were maintained at ambient temperature. The process conditions used in each example and the properties of the resulting composite sheets are set forth in Table 4 below.

Table 4

<u>EXAMPLE NUMBER.</u>	17 (3706)	18 (3707)	19 (3708)	20 (3709)	21 (3710)
<u>Process Conditions</u>					
Basis Weight - Nonwoven (g/m <sup>2</sup> )	28.3	19.8	14.2	9.9	28.3
Line speed (m/min)	36.6	39.6	42.7	45.7	36.6
Extruded melt temp - Layer 1 (°C)	233	233	233	233	233
Extruder speed - Layer 1 (RPM)	40	40	40	40	40
Extruded melt temp - Layer 2 (°C)	232	232	232	232	232
Extruder speed - Layer 2 (RPM)	33	33	33	33	33
Die temperature (°C)	216	216	216	216	216
Cylinder pressure (kPa)	207	207	207	207	207
Embossing roll temperature (°C)	43.3	43.3	43.3	43.3	43.3
Water bath temperature (°C)	19.4	19.4	19.4	19.4	19.4
<u>Composite Properties</u>					
Film Thickness (microns)	22	22	22	22	22
Composite Thickness (microns)	188	150	107	89	371
MVTR -(Desiccant Method) (g/m <sup>2</sup> /day)	3200	3400	3200	3100	3000
Dynamic Impact (g/m <sup>2</sup> @ 2400 J/m <sup>2</sup> )	0.0	0.25	0.19	0.28	0.52
Peel Strength (N/cm)					
MD	0.98	1.08	0.97	0.74	0.09
CD	0.69	0.53	0.34	0.45	0.08
Tensile Strength (N/cm)					
MD	7.0	6.3	5.6	--	--
CD	1.6	1.3	1.1	--	--
Elongation (%)					
MD	24.0	28.7	27.6	--	--
CD	53.4	61.7	51.0	--	--
Pinhole Seepage	0	0	0	0	0
Hydrostatic Head (cm)	198	183	231	203	211
Gurley Hill Air Porosity (sec)	>3600	>3600	>3600	>3600	>3600

- 5 Three samples of the composite sheet material (fibrous substrate and film without the polyethylene film) produced in Example 17 were tested according to the Viral Barrier test method described above. All three samples passed the Viral Barrier test (zero PFU/ml were detected after the 24 hour test period).

Examples 17-20 demonstrate that outstanding peel strength can be obtained at low bonding pressures, even in the absence of an adhesive. These Examples show that the presence of a limited amount of polyester in the fibrous substrate greatly enhances peel strength. These Examples also show that it is possible to achieve good peel strength and excellent dynamic impact barrier properties at the same time. Example 21 demonstrates that the lack of polyester results in low peel strength in the absence of an adhesive.

#### EXAMPLES 22-25

The film compositions used in Examples 22-25 had the following compositions:

<u>EXAMPLE</u>	22 (3644)	23 (3645)	24 (3646)	25 (3643)
<u>Film Layer 1</u> (wt. % of layer 1)				
Hytrel® 8206 (%)	—	40	—	—
Hytrel® 4778 (%)	—	60	100	—
Hytrel® 3548 (%)	100	—	—	—
<u>Film Layer 2</u> (wt. % of Layer 2)				
Hytrel® 8171 (%)	48	48	48	48
Hytrel® 8206 (%)	46	46	46	46
TiO <sub>2</sub> Concentrate (%)	6	6	6	6
Layer 1 (wt % of total film)	17	17	17	0
Layer 2 (wt % of total film)	83	83	83	100

A polymer film comprised of the Film Layers 1 and 2 set forth above was extrusion coated on a fibrous polypropylene nonwoven sheet by the following process. The nonwoven sheet was the carded polypropylene sheet material described for Examples 1-5 above.

The components for Film Layer 1 were mixed and fed in pellet form into a 4 inch (10.2 cm) diameter screw extruder that was connected to a melt combining block. The components for Film Layer 2 were also mixed and fed in pellet form into a different 3 inch (7.6 cm) diameter screw extruder that was connected to the same melt combining block. The components for Film Layers 1 and 2 were melted and coextruded to the melt combining block. The two layer melt was then fed to a die opening (0.76 mm microns by 102 cm) in a heated die

block. A bicomponent film with Film Layer 1 and Film Layer 2 was extruded from the die opening and was coated on the polypropylene nonwoven sheet without the application of an adhesive. The polypropylene fiber sheet was spaced 22.9 cm ( 9 in) below the opening of the die.

5           The polypropylene fiber sheet, the adhesive and the film were passed through a pair of nip rolls, as shown in Figure 4. The nip rolls were subjected to cylinder pressures of 689 kPa (100 psi). The process conditions used in each example and the properties of the resulting composite sheets are set forth in Table 5 below.

10           The film side of the composite sheet was subsequently glued to a polyethylene film to determine whether the composite sheet would remain bonded to such films under conditions that might be encountered in an absorbent article. A pressure sensitive linear SIS hotmelt adhesive (hotmelt adhesive H2031 from Ato Findley Adhesives, Inc., of Wauwatosa, Wisconsin) was applied to the film  
15           side of the composite sheet at an adhesive basis weight of 0.009 g/in<sup>2</sup> (13.95 g/m<sup>2</sup>) using a Meltex EP34s spray application head available from Nordson Corporation. The temperature of the adhesive leaving the spray head was 166°C.

          A sheet of polyethylene film with a thickness of 1.2 mil (30.5 microns) was pressed against the side of the composite sheet to which the adhesive was applied  
20           using a hand roller. The dry and wet strength of the bond between the composite sheet and the polyethylene film was measured and is reported in Table 5 below.

Table 5

<u>EXAMPLE NUMBER.</u>	22 (3644)	23 (3645)	24 (3646)	25 (3643)
<u>Process Conditions</u>				
Line speed (m/min)	27.4	27.4	40.8	27.4
Extruded melt temp - Layer 1 (°C)	243	243	243	243
Extruder speed - Layer 1 (RPM)	20	20	37	80
Extruded melt temp - Layer 2 (°C)	260	260	260	—
Extruder speed - Layer 2 (RPM)	21	21	27	—
Die temperature (°C)	243	243	243	243
Cylinder pressure (kPa)	689	689	689	689
Embossing roll temperature (°C)	60	60	60	60
Water bath temperature (°C)	49	49	49	49
<u>Composite Properties</u>				
Film Thickness (microns)	23	23	23	23
MVTR -(Desiccant Method) (g/m <sup>2</sup> /day)	3850	3150	2950	3600
Peel Strength (N/cm)				
MD	0.36	0.43	0.64	0.25
CD	0.25	0.46	0.51	0.20
Tensile Strength (N/cm)				
MD	12.4	12.4	13.1	—
CD	2.3	2.4	2.4	—
Elongation (%)				
MD	81	86	88	—
CD	105	107	105	—
Gurley Hill Air Porosity (sec)	>3600 sec	>3600 sec	>3600 sec	>3600 sec
Hydrostatic Head (cm)	66	46	56	43
<u>Article Construction</u>				
Dry Bond Strength (g/cm)	156.7	122.0	224.4	112.6
Wet Bond Strength (g/cm)	4.3	10.2	170.5	1.6

- 5 Examples 22-25 demonstrate that changes in the composition of a two layer moisture vapor permeable has a substantial impact on the MVTR of the composite sheet. These Examples also show that a polyethylene film can be bonded to the film layer of the composite sheet with conventional hot melt adhesives. Finally, these Examples show that by making the outer film layer from



a more hydrophobic polyether ester elastomer, good wet bond strength can be obtained (Example 24).

Examples 22-25 illustrate that the polypropylene based fibrous substrate requires > 3 times the bonding pressure relative to examples 17-20 which comprised polyester and polypropylene fiber blends. The compatible polyester fibers allow significantly lower pressures to be used while achieving a significant improvement in peel strength versus Examples 22-25. Examples 22-25, even though they were bonded at higher pressure, resulted in peel strengths that were nominally 50% of the peel achieved with examples 17-20. In order for Examples 22-25 to achieve the level of bonding seen with Examples 17-20, higher bonding pressure is required. Because Examples 22-25 already incorporate higher melt temperature which is amenable to bonding, but reduces the viscosity of the polymer, it is very likely that increasing the bonding strength will result in an increase in pinholes and impact related leakage.

15

#### EXAMPLES 26-29

A copolyether ester film was extrusion coated directly onto a number of different fibrous nonwoven sheets without the use of a separate adhesive. In Examples 26-29, the composition of the nonwoven sheet had a basis weight of 14.2 g/m<sup>2</sup> (0.5 oz/m<sup>2</sup>), and had one of the following compositions:

Composition A: a 50/50 blend of polyester staple fiber (Dacron® Type 54 polyester fiber manufactured by DuPont) and polypropylene staple fibers (Danaklon Hy Comfort polypropylene fiber manufactured by Danaklon America, Inc. of Athens, Georgia). The polyester and polypropylene staple fibers had fiber lengths of about 40 mm and a denier of 2. The fibers were carded and thermobonded on a B.F. Perkins Calender Bonder at a temperature of 130°-145° C with a very light nip pressure.

Composition B: a 50/50 blend of polyester and polypropylene fibers like Composition A with the exception the Dacron® Type 54 polyester fiber was replaced with shaped polyester fibers having a scalloped-oval cross-section, as described in U.S. Patent No. 3,914,488, and having an average fiber length of 40 mm and a denier of 1.4. The fibers were carded and thermobonded on a B.F. Perkins Calender Bonder at a temperature of 130°-145° C with a very light nip pressure.

Composition C: a blend of 75% polyester staple fiber (Dacron® Type 54 polyester fiber manufactured by DuPont) and 25% polypropylene

5 staple fibers (Danaklon Hy Comfort polypropylene fiber manufactured by Danaklon America, Inc. of Athens, Georgia). The polyester and polypropylene staple fibers had fiber lengths of about 40 mm and a denier of 2. The fibers were carded and thermobonded on a B.F. Perkins Calender Bonder at a temperature of 130°-145° C with a very light nip pressure.

10 Composition D: a 75/25 blend of polyester and polypropylene fibers like Composition C with the exception the Dacron® Type 54 polyester fiber was replaced with shaped polyester fibers having a scalloped-oval cross-section, as described in U.S. Patent No. 3,914,488, and having an average fiber length of 40 mm and a denier of 1.4. The fibers were carded and thermobonded on a B.F. Perkins Calender Bonder at a temperature of 130°-145° C with a very light nip pressure.

15 The nonwoven was laminated with a polymer film comprised of 48% Hytrel® 8206 copolyether elastomer, 48% Hytrel® 8171 copolyether elastomer, and 4% titanium dioxide. The copolyether ester polymer was fed in pellet form into a 38 mm diameter screw extruder that was connected to a heated die. The polymer was melted and then fed to a 36 cm by 250 micron die opening in the  
20 heated die block. The polymer melt was extruded from the die opening and was coated on the polypropylene nonwoven sheet as shown in Figure 3. The film was joined to the fibrous nonwoven sheet in a nip that was spaced about 10 cm from the die opening. The nip arrangement was like that of Examples 1-5, and the pressure cylinders were maintained at a constant pressure of about 140 kPa  
25 (20 psi). The process conditions used in each example and the properties of the resulting composite sheets are set forth in Table 6 below.

Table 6

<u>EXAMPLE NUMBER.</u>	26 (90E3)	27 (90F8)	28 (90G12)	29 (90G13)
<u>Nonwoven Composition</u>	A	B	C	D
<u>Process Conditions</u>				
Nonwoven speed (m/min)	12	12	12	12
Extruded melt temp (°C)	220	220	220	220
Extruder speed (RPM)				
Die temperature (°C)	220	220	220	220
Cylinder pressure (kPa)	140	140	140	140
<u>Composite Properties</u>				
Film Thickness (microns)	26	24	25	27
Composite Thickness (mm)				
MVTR -(Desiccant Method) (g/m <sup>2</sup> /day)	2600	3100	2400	2900
Dynamic Impact (g/m <sup>2</sup> @ 2400 J/m <sup>2</sup> )	0.19		0.00	0.00
Peel Strength (N/cm)				
MD	0.61	0.88	weak	0.63
CD	0.24	0.20	weak	0.11

Examples 26-29 illustrate the impact of the use of shaped fibers in the fibrous substrate of the composite structure. Without wishing to be bound by theory, it is believed that the shaped fibers, when brought into contact with the film melt, increase the surface area of the film, thereby increasing the flux of vapor through the composite structure. The types of non-porous, liquid impermeable, vapor permeable films transmit vapor by first absorption, then diffusion and finally evaporation. Increasing the surface area for the evaporation step will increase the vapor transmission.

#### EXAMPLES 30-41

Copolyether ester films were extrusion coated directly onto four different fibrous nonwoven sheets without the use of a separate adhesive. The four nonwovens were comprised of various combinations of polypropylene fibers and polyester fibers. The nonwoven sheets used in Examples 30-41 had one of the following compositions:

Composition E: carded polypropylene fibers thermally bonded as fully described in Examples 1-5

Composition F: blend of 26% polyethylene terephthalate polyester fibers (Dacron® Type 54 polyester fiber manufactured by DuPont) and 74% polypropylene fibers. blend of polyester staple fiber (Danaklon Hy Comfort polypropylene fiber manufactured by Danaklon America, Inc. of Athens, Georgia). The polyester and polypropylene staple fibers had fiber lengths of about 40 mm and a denier of 2. The fibers were carded and thermobonded on a B.F. Perkins Calender Bonder at a temperature of 130°-145° C with a very light nip pressure.

Composition G: a 50/50 blend of polyethylene terephthalate polyester fibers and polypropylene staple fibers identical to Composition A described above with respect to Examples 26-29.

Composition H: 100% polyethylene terephthalate polyester fibers with a fiber lengths of about 40 mm and a denier of 2. The fibers were carded and thermobonded on a B.F. Perkins Calender Bonder at a temperature of 130°-145° C with a very light nip pressure.

The two films used were as follows:

Film 1 was a single layer film comprised of 47% Hytrel® 8206 copolyether elastomer, 47% Hytrel® 8171 copolyether elastomer, and 6% titanium dioxide.

Film 2 was a two layer film in which Layer 1 was comprised of the blend of Film 1, and Layer 2 was comprised of 100% Hytrel® 8206 copolyether elastomer.

The copolyether ester polymer was fed in pellet form into a 38mm diameter screw extruder that was connected to a heated die. The polymer was melted and then fed to a 36 cm by 250 micron die opening in the heated die block. The polymer melt was extruded from the die opening and was extrusion coated on the nonwoven sheet without the application of an adhesive. The film was joined to the fibrous nonwoven sheet in a nip that was spaced about 10 cm from the die opening. The nip arrangement was like that of Examples 1-5, and the pressure cylinders were maintained at a constant pressure of about 140 kPa (20 psi).

The process conditions used in each example and the properties of the resulting composite sheets are set forth in Table 7 below.

Table 7

<u>EXAMPLE NUMBER.</u>	30 (130T1)	31 (130T4)	32 (130T3)	33 (130T2)
<u>Nonwoven Composition</u>	E	F	G	H
<u>Film Composition</u>	1	1	1	1
<u>Process Conditions</u>				
Line speed (m/min)	12.2	12.2	12.2	12.2
Extruded melt temp (°C)	220	220	220	220
Extruder speed (RPM)	20	20	20	20
Die temperature (°C)	220	220	220	220
Cylinder pressure (kPa)	138	138	138	138
<u>Composite Properties</u>				
Film Thickness (microns)	25	25	25	25
MVTR -(Desiccant Method) (g/m <sup>2</sup> /day)	4000	3700	3600	2900

Table 7 (continued)

<u>EXAMPLE NUMBER.</u>	34 (130V1)	35 (130V4)	36 (130V3)	37 (130V2)
<u>Nonwoven Composition</u>	E	F	G	H
<u>Film Composition</u>	1	1	1	1
<u>Process Conditions</u>				
Line speed (m/min)	21	21	21	21
Extruded melt temp (°C)	220	220	220	220
Extruder speed (RPM)	20	20	20	20
Die temperature (°C)	220	220	220	220
Cylinder pressure (kPa)	138	138	138	138
<u>Composite Properties</u>				
Film Thickness (microns)	12	12	12	12
MVTR -(Desiccant Method) (g/m <sup>2</sup> /day)	4400	4200	4500	3500

Table 7 (continued)

5

<u>EXAMPLE NUMBER.</u>	38 (130A1)	39 (130A4)	40 (130A3)	41 (130A2)
<u>Nonwoven Composition</u>	E	F	G	H
<u>Film Composition</u>	2	2	2	2
<u>Process Conditions</u>				
Line speed (m/min)	10.5	10.5	10.5	10.5
Extruded melt temp - layer 1 (°C)	220	220	220	220
Extruded melt temp - layer 2 (°C)	220	220	220	220
Extruder speed - layer 1 (RPM)	15	15	15	15
Extruder speed - layer 2 (RPM)	12	12	12	12
Die temperature (°C)	220	220	220	220
Cylinder pressure (kPa)	138	138	138	138
<u>Composite Properties</u>				
Film Thickness (microns)	25	25	25	25
MVTR -(Desiccant Method) (g/m <sup>2</sup> /day)	3500	2500	2900	1600

Examples 17-20 illustrated the positive impact of polyester, compatible, fiber inclusion in the fibrous substrate upon the peel strength. Examples 30-41 illustrate that the inclusion of polyester fibers can also have a negative affect on MVTR. This negative affect is associated with the increase in fiber to film bonds associated with the polyester, compatible, fibers. The data in Table 7 shows that the composite structure comprising a polypropylene only fibrous substrate has a higher MVTR than all but one of the other Examples, Example 36. The Table 7 data also shows that the composite structures with the blended, 50% polyester and 50% polypropylene, fibrous substrates are substantially higher in MVTR than the composite structure comprising a 100% polyester fibrous substrate.

#### EXAMPLE 42

A sample of Exxon Exxair XFB-100W microporous film, available from Exxon Chemical Company of Buffalo Grove, Illinois, USA, was laminated to a moisture vapor permeable film comprised of 100% Hytrel® 8171 copolyether elastomer. The microporous film was coated with the pressure sensitive linear SIS hotmelt adhesive discribed in Examples 1-5 above at an adhesive basis weight of .001 g/in<sup>2</sup>. The moisture vapor permeable film, which had been melt cast on a release paper with a thickness of 13 microns, was bonded to the adhesive on the microporous film.

The microporous sheet, the moisture vapor permeable film, and the composite of the two had the following properties.

	MVTR (g/m <sup>2</sup> /day) (Desiccant Method)	Dynamic Impact (g/m <sup>2</sup> @ 2400 J/m <sup>2</sup> )
Microporous Film	4,600	1.26
Hytrel® Film	3,900	0.00
Composite	3,500	0.23

This Example shows that the addition of a moisture vapor permeable film to a microporous film significantly reduces moisture impact related leakage from a microporous film.

#### COMPARATIVE EXAMPLE 1

A sample of Exxon Exxair XFB-100W microporous film, available from Exxon Chemical Company of Buffalo Grove, Illinois, USA, was tested for moisture vapor transmission rate, dynamic fluid transmission, microbial barrier

for sterile packaging, and liquid moisture seepage. The properties measured were as follows:

MVTR ( $\text{g/m}^2/24 \text{ hr}$ )	4000
Dynamic Impact ( $\text{g/m}^2 @ 2400 \text{ J/m}^2$ )	0.97
Microbial Barrier	Bacillus subtilis bacteria passage recorded in six of six samples tested after 15 minute exposure. (38.6 cm Hg vacuum; 2.8 l/min flow rate)
Moisture Seepage	Dye apparent on blotter indicating passage of liquid.

5           It will be apparent to those skilled in the art that modifications and variations can be made in breathable composite sheet material of this invention. The invention in its broader aspects is, therefore, not limited to the specific details or the illustrative examples described above. Thus, it is intended that all matter contained in the foregoing description, drawings and examples shall be interpreted  
10 as illustrative and not in a limiting sense.



## WHAT IS CLAIMED IS:

1. A moisture vapor permeable, substantially liquid impermeable composite sheet material comprising:

5 a fibrous nonwoven substrate, said substrate having opposite first and second planar sides;

a moisture vapor permeable thermoplastic film bonded to the first side of said fibrous substrate, said moisture vapor permeable film having an average  
10 thickness of less than 25 microns;

said composite sheet exhibiting a peel strength of at least 0.1 N/cm, a dynamic fluid transmission of less than about 0.75 g/m<sup>2</sup> when subjected to an impact energy of about 2400 joules/m<sup>2</sup>, a hydrostatic head of at least 60 cm, and  
15 having a moisture vapor transmission rate, according to the desiccant method, of at least 2800 g/m<sup>2</sup>/24hr.

2. The composite sheet of claim 1 wherein said moisture vapor permeable film is comprised of at least about 50% by weight of polymer selected  
20 from the group of block copolyether esters, block copolyether amides, polyurethanes, polyvinyl alcohol, and combinations thereof.

3. The composite sheet of claim 2 wherein said film has an average thickness of less than 20 microns.  
25

4. The composite sheet of claim 3 wherein said composite sheet exhibits a hydrohead of at least 120 cm, a dynamic fluid transmission of less than about 0.65 g/m<sup>2</sup> when subjected to an impact energy of about 2400 joules/m<sup>2</sup>, and a moisture vapor transmission rate, according to the dessicant method, of at least  
30 3200 g/m<sup>2</sup>/24hr.

5. The composite sheet of claim 4 wherein said film of said sheet has an average thickness of less than 15 microns, and the basis weight of said fibrous substrate is between about of 13.5 and about 40 g/m<sup>2</sup>.  
35

6. The composite sheet of claim 5 wherein said film of said sheet has an average thickness of less than 10 microns and a dynamic fluid transmission of less than about 0.5 g/m<sup>2</sup> when subjected to an impact energy of about

2400 joules/m<sup>2</sup>, and a moisture vapor transmission rate, according to the dessicant method, of at least 3800 g/m<sup>2</sup>/24hr.

7. The composite sheet of claim 3 wherein said composite sheet  
5 exhibits a hydrostatic head of at least 120 cm and a dynamic fluid transmission of less than about 0.5 g/m<sup>2</sup> when subjected to an impact energy of about 2400 joules/m<sup>2</sup>.

8. The composite sheet of claim 3 wherein the composite sheet is  
10 substantially free of micropores, and substantially no liquid moisture passes through the sheet when tested according to the liquid moisture seepage test.

9. The composite sheet of claim 8 wherein said composite sheet  
15 prevents passage of microbes when tested according to the ISO 11607 standard for sterile packaging materials.

10. The composite sheet of claim 9 wherein said composite sheet,  
when tested according to ASTM F1671, prevents the passage of microbe with a  
diameter greater than 0.025 microns.

20

11. The composite sheet of claim 3 wherein said moisture vapor  
permeable film consists essentially of a copolyether ester elastomer, and wherein  
said fibrous substrate consists essentially of a blend of between 20% and 80% by  
weight polyolefin polymer fibers and between 20% and 80% by weight polyester  
25 polymer fibers.

12. The composite sheet of claim 11 wherein said polyester fibers are  
shaped fibers with a scalloped-oval cross-section and wherein the composite sheet  
has and a moisture vapor transmission rate, according to the dessicant method, of  
30 at least 3000 g/m<sup>2</sup>/24hr.

13. The composite sheet of claim 3 wherein said moisture vapor  
permeable film is bonded to the fibrous substrate with a hotmelt adhesive applied  
between the fibrous nonwoven substrate and the moisture vapor permeable film at  
35 an adhesive basis weight between 0.5 and 5 mg/in<sup>2</sup>, said adhesive contacting less  
than 75% of the surface of the first side of the fibrous substrate.

14. The composite sheet of claim 13 wherein said fibrous substrate consists essentially of polyolefin polymer fibers, and wherein said adhesive is applied between said fibrous substrate and said moisture vapor permeable film at a weight of between 1 and 3 mg per 6.45 cm<sup>2</sup> of the first surface of the fibrous substrate.

15. The composite sheet of claim 3 wherein said moisture vapor permeable film has first and second layers, each of said layers being comprised of a different moisture vapor permeable thermoplastic polymer composition.

10

16. The composite sheet of claim 15 wherein

said first layer of said moisture vapor permeable film comprises at least 60% of the total weight of the film and is comprised of a substantially hydrophilic layer,

15

said second layer of said moisture vapor permeable film comprises a substantially hydrophobic layer, and

20

said first layer of said moisture vapor permeable film abuts said fibrous substrate.

17. The composite sheet of claim 16, wherein said composite sheet further includes an additional layer of diverse construction adhesively bonded to the second layer of said moisture vapor permeable film.

25

18. The composite sheet of claim 17 wherein said composite sheet had a wet bond strength of at least 80 g/cm.

30

19. The composite sheet of claim 17, wherein said additional layer comprises a microporous film.

20. A method for making a moisture vapor permeable, substantially liquid impermeable composite sheet material comprising a fibrous nonwoven substrate and a moisture vapor permeable thermoplastic film, said method comprising the steps of:

35

(a) providing a nonwoven substrate;

(b) melt extruding the moisture vapor permeable film through a flat film die onto said nonwoven substrate to form a composite sheet material, said nonwoven substrate including fibers which are incompatible with said moisture vapor permeable film;

- 5 (c) subjecting said composite sheet material to a confining pressure by passing said composite sheet material through a nip; and  
(d) collecting the sheet material onto a roll.

10 21. The method of Claim 20, wherein an adhesive is applied to said nonwoven substrate before said moisture vapor permeable film is extruded onto said nonwoven substrate.

15 22. The method of Claim 20, wherein said moisture vapor permeable film comprises a multilayer film.

23. The method of Claim 20, wherein said substrate includes fibers which are compatible with said moisture vapor permeable film.

20 24. A moisture vapor permeable, substantially liquid impermeable sheet material made according to the method of Claim 20.

1/3

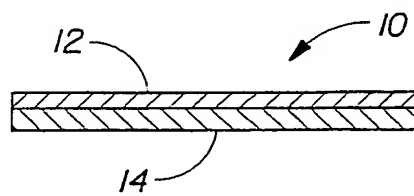


Fig. 1

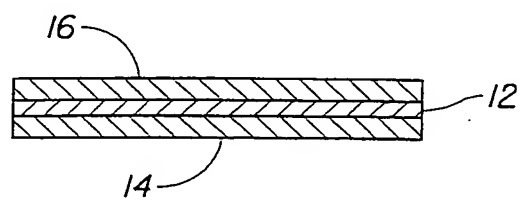


Fig. 2

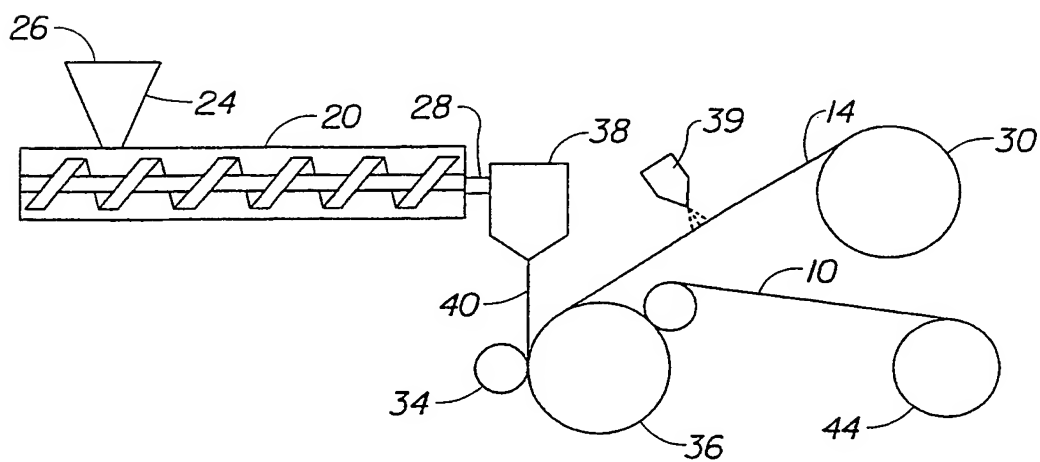


Fig. 3

2/3

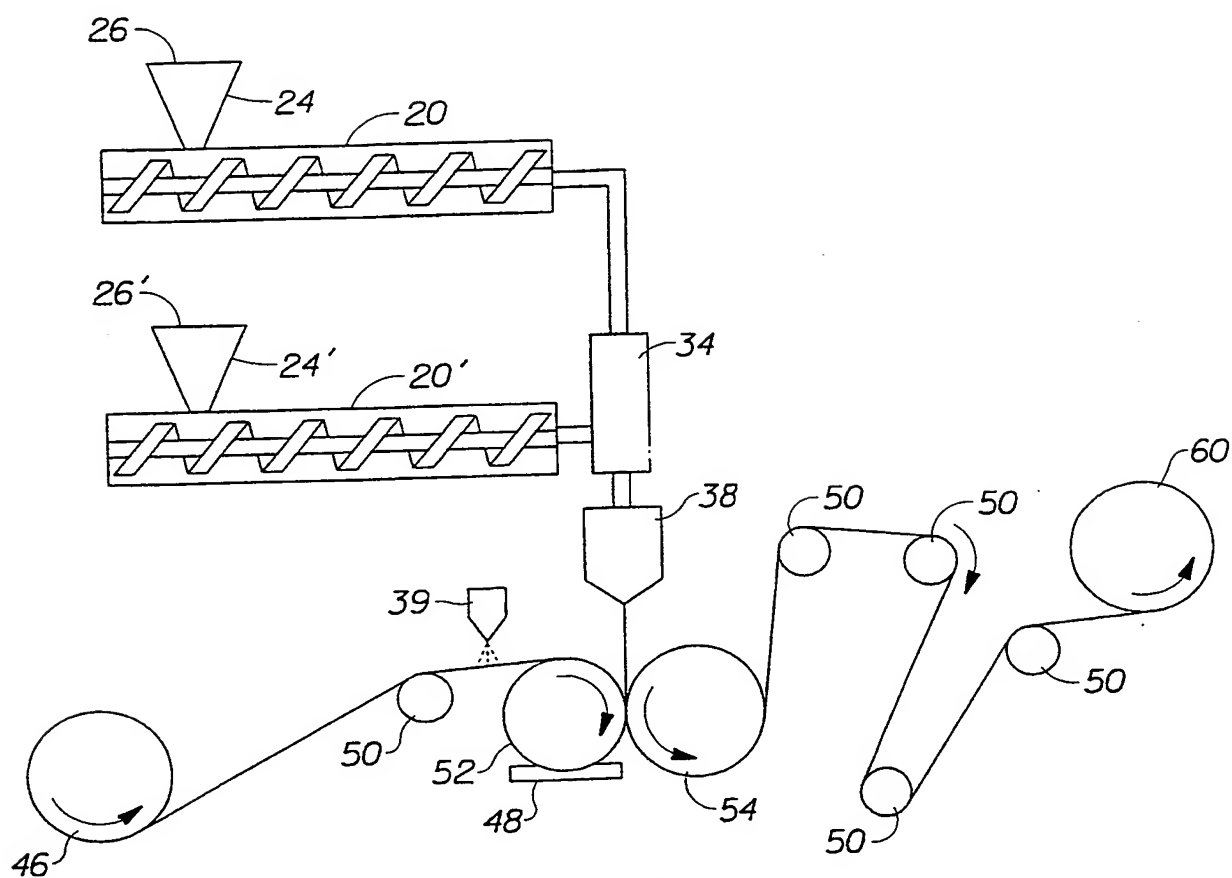


Fig. 4

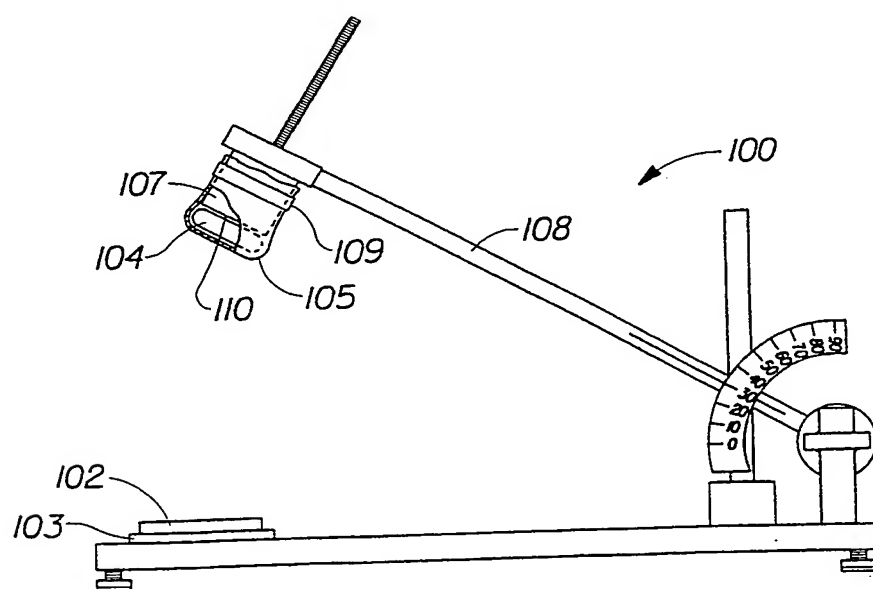


Fig. 5

3/3

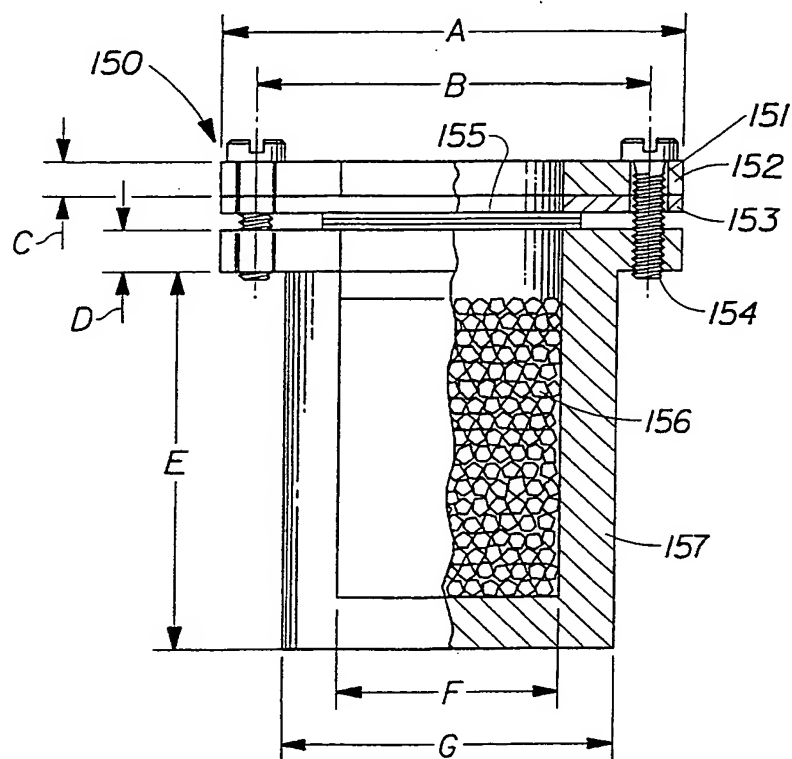


Fig. 6

# INTERNATIONAL SEARCH REPORT

International Application No

PC1, JS 98/25426

A. CLASSIFICATION OF SUBJECT MATTER  
IPC 6 B32B27/12 D04H13/00

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)  
IPC 6 B32B

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
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P,X

WO 97 45259 A (CURRO JOHN JOSEPH  
;OSTAPCHENKO GEORGE JOSEPH (US); VAIDYA  
SHAILAJA) 4 December 1997  
see page 3, line 23 - line 31; claims  
1,8,9; examples; tables 1-3  
---

1-24

X

EP 0 688 826 A (ATOCHM ELF SA)  
27 December 1995  
see page 2, line 34 - page 5, line 49  
see page 7  
see claims  
---

1-10,20,  
24

X

EP 0 560 630 A (MCNEIL PPC INC)  
15 September 1993  
see claims 1,2,9-15; example 2  
---

1-10,20,  
24

-/--



Further documents are listed in the continuation of box C.



Patent family members are listed in annex.

### \* Special categories of cited documents :

- \*A\* document defining the general state of the art which is not considered to be of particular relevance
- \*E\* earlier document but published on or after the international filing date
- \*L\* document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
- \*O\* document referring to an oral disclosure, use, exhibition or other means
- \*P\* document published prior to the international filing date but later than the priority date claimed

\*T\* later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

\*X\* document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

\*Y\* document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.

\*Z\* document member of the same patent family

Date of the actual completion of the international search

8 March 1999

Date of mailing of the international search report

30.03.99

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Authorized officer

De Jonge, S



# INTERNATIONAL SEARCH REPORT

International Application No

PC1, US 98/25426

## C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	<p>WO 95 23696 A (GRACE W R &amp; CO)</p> <p>8 September 1995</p> <p>see claims 1,9-13,16</p> <p>see table 1</p> <p>sample 6</p> <p>-----</p>	1,2,24

# INTERNATIONAL SEARCH REPORT

International application No.  
PCT/US 98/25426

## Box I Observations where certain claims were found unsearchable (Continuation of item 1 of first sheet)

This International Search Report has not been established in respect of certain claims under Article 17(2)(a) for the following reasons:

1. ☐ Claims Nos.:  
because they relate to subject matter not required to be searched by this Authority, namely:
2. ☐ Claims Nos.:  
because they relate to parts of the International Application that do not comply with the prescribed requirements to such an extent that no meaningful International Search can be carried out, specifically:
3. ☐ Claims Nos.:  
because they are dependent claims and are not drafted in accordance with the second and third sentences of Rule 6.4(a).

## Box II Observations where unity of invention is lacking (Continuation of item 2 of first sheet)

This International Searching Authority found multiple inventions in this international application, as follows:

see additional sheet

1. ☐ As all required additional search fees were timely paid by the applicant, this International Search Report covers all searchable claims.
2. ☒ As all searchable claims could be searched without effort justifying an additional fee, this Authority did not invite payment of any additional fee.
3. ☐ As only some of the required additional search fees were timely paid by the applicant, this International Search Report covers only those claims for which fees were paid, specifically claims Nos.:
4. ☐ No required additional search fees were timely paid by the applicant. Consequently, this International Search Report is restricted to the invention first mentioned in the claims; it is covered by claims Nos.:

Remark on Protest

- ☐ The additional search fees were accompanied by the applicant's protest.
- ☐ No protest accompanied the payment of additional search fees.

FURTHER INFORMATION CONTINUED FROM PCT/ISA/ 210

This International Searching Authority found multiple (groups of) inventions in this international application, as follows:

1. Claims: 1-19

composite material comprising a fibrous nonwoven substrate, and a moisture vapor permeable thermoplastic film with a thickness of less than 25 microns, characterised by some properties expressed in specific parameters

2. Claims: 20-24

method for making a composite material comprising a fibrous nonwoven substrate and a moisture vapor permeable film, and composite materials obtained by this method

# INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No

PC1, JS 98/25426

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
WO 9745259 A	04-12-1997	US 5865823 A AU 3371697 A AU 5168898 A WO 9819861 A	02-02-1999 05-01-1998 29-05-1998 14-05-1998
EP 0688826 A	27-12-1995	FR 2721320 A AU 673646 B AU 2172195 A CA 2152034 A CN 1118737 A DE 19522333 A FI 953025 A JP 2696788 B JP 8003329 A NO 952375 A SG 33383 A US 5869414 A US 5800928 A	22-12-1995 14-11-1996 18-01-1996 21-12-1995 20-03-1996 21-12-1995 21-12-1995 14-01-1998 09-01-1996 21-12-1995 18-10-1996 09-02-1999 01-09-1998
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